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Supporting Information

for

A cyanine dimerization rendering positive cooperativity in molar absorptivity

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UV–vis absorption and fluorescence emission spectra; fluorescence lifetime spectra; $^{1}H/^{13}C$ NMR, and HRMS spectra

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Table of contents

Page	Contents	
S2	General materials and instruments	
S2	Experimental methods	
S3	Synthetic procedures and compound characterization	
S8–S10	Figures S1–S5	UV–vis absorption spectra and fluorescence emission spectra of dyes.
S10	Figure S6	Fluorescence lifetime spectra of dyes.
S11–S40	Figures S7–S36	^{1}H NMR, ^{13}C NMR and HRMS spectra of all compounds.
S41	References	

General materials and instruments

All organic solvents used in this study were commercially available and of analytical grade. Starting materials **1**, **4**, **5**, and **10** were obtained from commercial suppliers. NMR spectra were recorded on a Bruker AV-400 spectrometer in DMSO-d_6 or $CDCl_3$. HRMS spectra were obtained on a Waters Xevo G2 TOF MS/ Waters GCT Premier/ ABS MALDI TOF MS spectrometer. UV–vis absorption spectra were measured on a SHIMADZU UV-2600i UV–vis spectrophotometer. Fluorescence emission spectra were recorded on a PTI-QM4 steady-state fluorimeter. Fluorescence lifetime spectra were determined on a custom-ordered HORIBA DeltaFLEX. Mouse vascular and lymphatic imaging were performed on a small animal system (MARS, Artemis Intelligent Imaging, Shanghai, China).

Experimental methods

Determination of molar absorptivity (ε):

The dye was dissolved in four different organic solvents at concentrations of 0.02, 0.04, 0.06, 0.08, and 0.10 μM, ensuring that the absorbance of each diluted solution remained below 0.1. According to the Beer–Lambert law, a linear relationship was established between the absorbance at λ_{max} and the dye concentration. With a cuvette path length of 1 cm, the molar absorptivity was obtained from the slope of the linear fit.

Determination of relative fluorescence quantum yields (Φ_F):

The relative fluorescence quantum yields of the monomer **M780** and dimers were measured using the absolute quantum yields of **ICG** and **ECY** as references, respectively (**ICG** in MeOH, Φ_F = 7.8%; **ECY** in $CHCl_3$, Φ_F = 19%). The absorbances of the dye samples and the reference were adjusted to fall within the range of 0.05–0.1 at the excitation wavelength. The Φ_F was finally calculated using the following equation:

$$\Phi_X = \Phi_S \, (A_S \, F_X / A_X \, F_S) \, (n_X / n_S)^2$$

Where Φ_S is the absolute quantum yield of the reference; A_X and A_S are the absorbances at the excitation wavelength; F_X and F_S are the corresponding integrated fluorescence intensities; n_X and n_S are the solvent refractive indexes; and the subscripts X and S denote the dye and the reference, respectively.

Determination of fluorescence lifetimes (τ):

The absorbance of the dye in CH_2Cl_2 was kept below 0.1. Pulsed semiconductor light sources with wavelengths close to the excitation maxima of the dyes (783 nm for monomers and 827 nm for dimers) were used to record the time-resolved fluorescence decay curves of the dye solutions and the scatterer solution (instrument response function, IRF). The fluorescence lifetimes were obtained by fitting the decay curves to a first-order exponential function.

In vivo imaging assay:

CyD7Bn NPs were successfully prepared according to literature procedures [1].

Female BALB/c mice (6–8 weeks old, body weight 20 ± 2 g) received **CyD7Bn** NPs via tail-vein injection (100 μL, 1 mg/kg) or local injection (20 μL, 0.2 mg/kg) into the hindlimb footpad, gluteal muscle, and forelimb footpad. Whole-body vasculature, hindlimb vasculature, isolated organs, and the lymphatic system were visualized by NIR in vivo imaging (excitation: 880 nm; long-pass filter: 1000–1350 nm).

All animal experiments were approved by the Animal Care and Use Committee of Shanghai University of Traditional Chinese Medicine (IACUC, approval number: PZSHUTCM2509050011) and conducted following institutional guidelines.

Synthetic procedures and compound characterization

Synthesis of compound 2:

4,4'-Diaminobiphenyl (**1**, 9.21 g, 50.00 mmol, 1.00 equiv) was dissolved in an aqueous solution of HCl (10 M, 180 mL). A solution of $NaNO_2$ (6.90 g, 100.00 mmol, 2.00 equiv) in H_2O (50 mL) was added portionwise at −20 °C, and the mixture was stirred at this temperature for 30 min. A preformed solution of selenium powder (160 mg, 2.00 mmol, 0.04 equiv) and $SnCl_2$ (37.92 g, 200.00 mmol, 4.00 equiv) in HCl (12 M, 50 mL) was then added dropwise. After the addition was complete, the ice bath was immediately removed, and the reaction was allowed to warm to room temperature and stirred for an additional 1 h. The precipitated solid was collected by vacuum filtration, washed three times with 3 M HCl, affording compound **2** as a tan powdery solid (10.7 g, 100%). The intermediate was used directly without further purification.

Synthesis of compound 3:

Compound **2** (10.71 g, 50.00 mmol, 1.00 equiv) was placed in a 100 mL round-bottomed flask, and anhydrous acetic acid (70 mL) and 3-methyl-2-butanone (53.17 mL, 500.00 mmol, 10.00 equiv) were added sequentially. The mixture was stirred at 120 °C under an argon atmosphere for 12 h. After completion of the reaction as monitored by TLC, the mixture was cooled to room temperature and adjusted to pH 6 with a saturated aqueous solution of $NaHCO_3$, then extracted with H_2O and CH_2Cl_2. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude product was further purified by silica gel column chromatography using PE/EA = 10:1 (v/v) as the final eluent, affording compound **3** as a brown powdery solid (10.8 g, yield: 68%). ^{1}H NMR (400 MHz, DMSO-d_6, ppm): δ 7.74 (d, J = 2.0 Hz, 2H), 7.57 (dd, J = 8.0, 1.9 Hz, 2H), 7.48 (d, J = 7.9 Hz, 2H), 2.23 (s, 6H), 1.31 (s, 12H).

Synthesis of compound M1 (General synthetic method A):

Compound **3** (9.49 g, 30.00 mmol, 1.00 equiv) in anhydrous MeCN (50 mL) and iodoethane (24.00 mL, 300.00 mmol, 10.00 equiv) was placed in a 250 mL thick-walled pressure vessel, and the mixture was stirred at 80 °C under an argon atmosphere for 12 h. After completion of the reaction as monitored by TLC, the mixture was cooled to room temperature and the precipitated solid was collected by vacuum filtration, washed three times with MeCN, affording compound **M1** as a tan powdery solid (10.0 g, 53%).^{1}H NMR (400 MHz, DMSO-d_6, ppm): δ 8.41 (s, 2H), 8.14 (d, *J* = 8.3 Hz, 2H), 8.08 (d, *J* = 8.4 Hz, 2H), 4.58 (q, *J* = 7.4 Hz, 4H), 2.90 (s, 6H), 1.66 (s, 12H), 1.49 (t, *J* = 7.2 Hz, 6H). ^{13}C NMR (100 MHz, DMSO-d_6, ppm): δ 197.0, 143.5, 141.2, 140.5, 128.5, 123.1, 116.4, 54.9, 43.8, 30.2, 22.5, 14.7, 13.3. HRMS-ESI (m/z): calculated for; $C_{26}H_{33}N_2^+$, $[M]^+$: 373.2638, found: 373.2639.

Synthesis of compound M2:

2,3,3-Trimethylindolenine (**4**, 16.05 mL, 100.00 mmol, 1.00 equiv), iodoethane (16.00 mL, 200.00 mmol, 2.00 equiv) were reacted according to general synthetic method A to afford compound **M2** as a purplish-red powdery solid (29.6 g, yield: 94%). ^{1}H NMR (400 MHz, DMSO-d_6, ppm): δ 8.01 – 7.98 (m, 1H), 7.89 – 7.84 (m, 1H), 7.65 – 7.59 (m, 2H), 4.52 (q, *J* = 7.3 Hz, 2H), 2.87 (s, 3H), 1.54 (s, 6H), 1.45 (t, *J* = 7.3 Hz, 3H).

Synthesis of compound M3:

2,3,3-Trimethyl-4,5-benzindole (**5**, 6.28 g, 30.00 mmol, 1.00 equiv), iodoethane (4.80 mL, 60.00 mmol, 2.00 equiv) were reacted according to general synthetic method A to afford compound **M3** as a tan powdery solid (9.5 g, yield: 87%).^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ 7.57 (d, *J* = 8.5 Hz, 1H), 7.48 (d, *J* = 8.8 Hz, 1H), 7.40 (d, *J* = 9.0 Hz, 1H), 7.29 (d, *J* = 8.9 Hz, 1H), 7.05 (ddd, *J* = 8.4, 6.9, 1.4 Hz, 1H), 6.95 (ddd, *J* = 8.2, 6.9, 1.2 Hz, 1H), 4.06 (s, 3H), 3.94 (q, *J* = 7.4 Hz, 2H), 1.09 (s, 6H), 0.89 (t, *J* = 7.4 Hz, 3H).

Synthesis of compound 6–9 and N1:

Compounds **6–9** and **N1** were synthesized according to literature procedures [2].

Synthesis of compound W1 (general synthetic method B):

Compound **M2** (2.52 g, 8.00 mmol, 1.00 equiv) and compound **N1** (1.66 g, 9.60 mmol, 1.20 equiv) were placed in a 250 mL thick-walled pressure flask, and anhydrous acetic acid (20 mL) and anhydrous toluene (20 mL) were added sequentially. The mixture was stirred at 110 °C under an argon atmosphere for 5 h. After completion of the reaction as monitored by TLC, the mixture was cooled to room temperature and concentrated under reduced pressure. The crude product was further purified by silica gel column chromatography using PE/EA= 80:1 (v/v) as the final eluent, affording compound **W1** as a pink powdery solid (1.8 g, yield: 67%). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 10.25 (s, 1H), 7.84 (d, *J* = 12.8 Hz, 1H), 7.22 (t, *J* = 7.6 Hz, 2H), 6.97 – 6.93 (m, 1H), 6.72 (d, *J* = 7.8 Hz, 1H), 5.52 (d, *J* = 12.7 Hz, 1H), 3.75 (q,

J = 7.1 Hz, 2H), 2.58 (t, *J* = 5.6 Hz, 2H), 2.49 (t, *J* = 6.1 Hz, 2H), 1.76 (d, *J* = 6.3 Hz, 2H), 1.66 (s, 6H), 1.28 (t, *J* = 7.2 Hz, 3H).

Synthesis of compound W2:

Compound **M3** (1.83 g, 5.00 mmol, 1.00 equiv), compound **N1** (1.04 g, 6.00 mmol, 1.20 equiv), anhydrous acetic acid (15 mL) and anhydrous toluene (15 mL) were reacted according to general synthetic method B to afford compound **W2** as a red powdery solid (1.5 g, yield: 79%). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 10.27 (s, 1H), 8.04 (d, *J* = 8.6 Hz, 1H), 7.97 (d, *J* = 13.0 Hz, 1H), 7.83 (d, *J* = 8.3 Hz, 1H), 7.78 (d, *J* = 8.7 Hz, 1H), 7.48 (t, *J* = 7.7 Hz, 1H), 7.32 – 7.27 (m, 1H), 7.09 (d, *J* = 8.7 Hz, 1H), 5.55 (d, *J* = 12.9 Hz, 1H), 3.85 (q, *J* = 7.3 Hz, 2H), 2.61 (t, *J* = 6.2 Hz, 2H), 2.50 (t, *J* = 6.2 Hz, 2H), 1.99 (s, 6H), 1.78 (q, *J* = 6.1 Hz, 2H), 1.36 – 1.31 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 190.8, 163.7, 148.8, 140.9, 131.4, 130.2, 129.9, 129.7, 129.0, 128.4, 127.0, 122.8, 121.7, 109.1, 92.1, 48.6, 38.7, 37.3, 27.5, 26.8, 24.6, 24.3, 21.0, 11.6. HRMS-ESI (m/z): calculated for; $C_{25}H_{26}ClNO$, $[M + H]^+$: 392.1776, found: 392.1782.

Synthesis of compound W3:

Compound **8** (977 mg, 3.00 mmol, 1.00 equiv), compound **N1** (621 mg, 3.60 mmol, 1.20 equiv), anhydrous acetic acid (10 mL) and anhydrous toluene (10 mL) were reacted according to general synthetic method B to afford compound **W3** as a red powdery solid (792 mg, yield: 55%). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 10.22 (s, 1H), 7.79 (d, *J* = 12.8 Hz, 1H), 6.99 (s, 1H), 6.54 (s, 1H), 5.58 (d, *J* = 12.8 Hz, 1H), 3.74 (q, *J* = 7.1 Hz, 2H), 2.58 (t, *J* = 6.3 Hz, 2H), 2.49 (t, *J* = 6.1 Hz, 2H), 2.18 (dq, *J* = 14.4, 7.1 Hz, 2H), 1.94 (dd, *J* = 13.8, 7.1 Hz, 2H), 1.78 (t, *J* = 6.2 Hz, 2H), 1.68 (s, 4H), 1.30 (s, 6H), 1.26 (s, 6H), 0.51 (t, *J* = 7.3 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 190.6, 158.9, 149.1, 144.3, 137.8, 131.5, 127.5, 121.7, 119.8, 103.6, 93.0, 57.0, 37.0, 35.2, 35.2, 34.7, 34.2, 34.1, 32.2, 32.1, 26.7, 24.6, 21.0, 11.2, 8.9. HRMS-ESI (m/z): calculated for; $C_{31}H_{42}ClNO$, $[M + H]^+$: 480.3028, found: 480.3034.

Synthesis of compound W4:

Compound **9** (1.12 g, 2.50 mmol, 1.00 equiv), compound **N1** (518 mg, 3.00 mmol, 1.20 equiv), anhydrous acetic acid (8 mL) and anhydrous toluene (8 mL) were reacted according to general synthetic method B to afford compound **W4** as a claret-red powdery solid (1.3 g, yield: 83%). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 10.30 (s, 1H), 7.07 – 6.97 (m, 8H), 6.77 – 6.73 (m, 4H), 6.20 (s, 1H), 5.41 (d, *J* = 12.9 Hz, 1H), 3.53 – 3.42 (m, 4H), 3.27 (q, *J* = 7.1 Hz, 2H), 2.61 (t, *J* = 6.3 Hz, 2H), 2.56 (t, *J* = 6.1 Hz, 2H), 1.84 (q, *J* = 6.2 Hz, 2H), 1.68 (s, 4H), 1.30 (s, 6H), 1.20 (s, 6H), 0.56 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 190.7, 157.7, 149.0, 145.0, 142.7, 137.0, 136.5, 132.3, 131.5, 130.6, 130.3, 127.9, 127.4, 126.3, 122.4, 122.2, 104.0, 93.7, 57.4, 45.8, 36.5, 35.2, 34.6, 34.2, 32.2, 32.1, 26.8, 24.7, 21.1, 10.1. HRMS-ESI (m/z): calculated for; $C_{41}H_{46}ClNO$, $[M + H]^+$: 604.3341, found: 604.3344.

Synthesis of compound CyD7 (General synthetic method C):

Compound **W1** (68 mg, 0.20 mmol, 2.00 equiv), compound **M1** (63 mg, 0.10 mmol, 1.00 equiv), and sodium acetate (16 mg, 0.20 mmol, 2.00 equiv) were sequentially placed in a 50 mL round-bottom flask, and acetic anhydride (10 mL) was added as the reaction solvent. The mixture was stirred at 130 °C under an argon atmosphere for 4 h. After completion of the reaction as monitored by TLC, the mixture was cooled to room temperature and concentrated under reduced pressure. The crude product was further purified by silica gel column chromatography using CH_2Cl_2/MeOH = 70/1 (v/v) as the final eluent, affording compound **CyD7** as a black powdery solid (45 mg, yield: 35%). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 8.38 (t, *J* = 12.8 Hz, 4H), 7.81 (s, 2H), 7.68 (d, *J* = 8.1 Hz, 2H), 7.38 (dd, *J* = 15.4, 8.0 Hz, 6H), 7.25 (d, *J* = 6.9 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 6.23 (dd, *J* = 14.1, 8.4 Hz, 4H), 4.32 – 4.20 (m, 8H), 2.73 (t, *J* = 6.5 Hz, 8H), 1.99 (s, 4H), 1.83 (s, 12H), 1.73 (s, 12H), 1.47 (q, *J* = 7.1 Hz, 12H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 171.9, 171.8, 150.5, 144.3, 142.4, 141.8, 141.3, 141.2, 137.9, 128.9, 127.8, 127.6, 127.4, 125.4, 122.4, 121.4, 111.4, 110.6, 101.5, 100.9, 49.7, 49.4, 40.1, 39.8, 28.2, 28.1, 26.6, 21.7, 20.8, 12.6, 12.4, 6.1. HRMS-ESI (m/z): calculated for; $C_{68}H_{78}Cl_2N_4^{2+}$, $[M/2]^+$: 510.2797, found: 510.2818.

Synthesis of compound CyD7.5:

Compound **W2** (78 mg, 0.20 mmol, 2.00 equiv), compound **M1** (63 mg, 0.10 mmol, 1.00 equiv), and sodium acetate (16 mg, 0.20 mmol, 2.00 equiv) were reacted according to general synthetic method C to afford compound **CyD7.5** as a black powdery solid (56 mg, yield: 41%). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 8.51 (d, *J* = 14.2 Hz, 2H), 8.36 (d, *J* = 13.7 Hz, 2H), 8.15 (d, *J* = 8.6 Hz, 2H), 7.98 (d, *J* = 8.7 Hz, 4H), 7.80 (s, 2H), 7.66 (t, *J* = 7.9 Hz, 4H), 7.50 (t, *J* = 8.9 Hz, 4H), 7.29 (d, *J* = 8.5 Hz, 2H), 6.32 (d, *J* = 14.3 Hz, 2H), 6.16 (d, *J* = 13.9 Hz, 2H), 4.43 (q, *J* = 7.3 Hz, 4H), 4.24 (q, *J* = 7.6 Hz, 4H), 2.82 – 2.77 (m, 4H), 2.74 (t, *J* = 6.1 Hz, 4H), 2.03 (s, 7H), 1.85 (s, 12H), 1.63 (s, 9H), 1.53 (d, *J* = 7.5 Hz, 6H), 1.49 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 199.2, 190.8, 161.7, 148.8, 143.5, 140.2, 139.4, 131.4, 128.4, 127.9, 122.9, 121.8, 120.9, 106.8, 92.7, 92.4, 46.6, 37.1, 28.3, 28.1, 26.7, 24.5, 20.9, 11.2. HRMS-ESI (m/z): calculated for; $C_{76}H_{82}Cl_2N_4^{2+}$, $[M/2]^+$: 560.2953, found: 560.3060.

Synthesis of compound CyD7Et:

Compound **W3** (96 mg, 0.20 mmol, 2.00 equiv), compound **M1** (63 mg, 0.10 mmol, 1.00 equiv), and sodium acetate (16 mg, 0.20 mmol, 2.00 equiv) were reacted according to general synthetic method C to afford compound **CyD7Et** as a purple-black powdery solid (45 mg, yield: 32%). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 8.40 (d, *J* = 14.2 Hz, 2H), 8.28 (d, *J* = 13.7 Hz, 2H), 7.68 (s, 2H), 7.63 (d, *J* = 10.0 Hz, 2H), 7.23 (t, *J* = 4.2 Hz, 4H), 7.07 (s, 2H), 6.42 (d, *J* = 14.3 Hz, 2H), 6.06 (d, *J* = 13.7 Hz, 2H), 4.35 (q, *J* = 7.2 Hz, 4H), 4.17 (q, *J* = 7.1 Hz, 4H), 2.78 (t, *J* = 6.3 Hz, 4H), 2.72 (t, *J* = 6.3 Hz, 4H), 2.23 (dq, *J* = 14.2, 6.8 Hz, 8H), 2.00 (t, *J* = 6.1 Hz, 4H), 1.81 (s, 12H), 1.72 (s, 8H), 1.47 (t, *J* = 7.2 Hz, 12H), 1.35 (s, 12H), 1.31 (s, 12H), 0.48 (t, *J* = 7.3 Hz, 12H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 171.1, 169.5, 149.9, 146.3, 144.6, 143.9, 142.0, 141.8, 141.6, 141.1, 137.0, 135.1, 127.6, 126.8, 121.0, 120.4, 110.4, 108.4,

103.5, 99.4, 60.5, 49.0, 40.6, 39.6, 35.0, 34.9, 34.8, 32.2, 28.3, 26.9, 26.6, 20.7, 13.1, 12.3, 8.7. HRMS-ESI (m/z): calculated for; $C_{88}H_{114}Cl_2N_4^{2+}$, $[M/2]^+$: 648.4205, found: 648.4205.

Synthesis of compound CyD7Bn:

Compound **W4** (121 mg, 0.20 mmol, 2.00 equiv), compound **M1** (63 mg, 0.10 mmol, 1.00 equiv), and sodium acetate (16 mg, 0.20 mmol, 2.00 equiv) were reacted according to general synthetic method C to afford compound **CyD7Bn** as a black powdery solid (78 mg, yield: 47%). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 8.75 (d, *J* = 14.2 Hz, 2H), 8.39 (d, *J* = 13.8 Hz, 2H), 7.75 (d, *J* = 1.9 Hz, 2H), 7.67 (dd, *J* = 8.3, 2.0 Hz, 2H), 7.32 – 7.28 (m, 6H), 7.08 – 7.02 (m, 10H), 6.67 (d, *J* = 7.0 Hz, 10H), 6.19 (dd, *J* = 14.0, 6.5 Hz, 4H), 4.29 – 4.22 (m, 4H), 3.81 (q, *J* = 7.1 Hz, 4H), 3.65 (d, *J* = 13.6 Hz, 4H), 3.57 (d, *J* = 13.5 Hz, 4H), 2.82 – 2.78 (m, 8H), 2.08 – 2.04 (m, 4H), 1.86 (s, 12H), 1.73 (s, 8H), 1.50 (t, *J* = 7.2 Hz, 6H), 1.35 (s, 12H), 1.25 (s, 12H), 0.74 (t, *J* = 7.2 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 170.4, 168.9, 149.9, 146.8, 144.8, 142.9, 142.7, 142.0, 141.5, 141.0, 137.4, 134.7, 134.0, 129.7, 127.9, 127.7, 127.5, 127.2, 122.8, 121.2, 110.9, 108.3, 103.6, 100.3, 60.5, 49.3, 46.2, 39.9, 39.7, 34.9, 34.8, 34.7, 32.1, 28.4, 26.9, 26.7, 20.8, 12.5, 11.8. HRMS-ESI (m/z): calculated for; $C_{108}H_{122}Cl_2N_4^{2+}$, $[M/2]^+$: 772.9543, found: 772.9535.

Synthesis of compound M780:

Compound **M2** (315 mg, 1.00 mmol, 2.00 equiv), compound **N1** (86 mg, 0.50 mmol, 1.00 equiv), and sodium acetate (82 mg, 1.00 mmol, 2.00 equiv) were reacted according to general synthetic method C to afford compound **M780** as a black powdery solid (278 mg, yield: 87%).^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 8.36 (d, *J* = 14.0 Hz, 2H), 7.40 (d, *J* = 7.5 Hz, 4H), 7.30 – 7.25 (m, 2H), 7.21 (d, *J* = 7.9 Hz, 2H), 6.21 (d, *J* = 14.1 Hz, 2H), 4.27 (q, *J* = 7.3 Hz, 4H), 2.75 (t, *J* = 6.2 Hz, 4H), 2.03 – 1.96 (m, 2H), 1.73 (s, 12H), 1.47 (t, *J* = 7.2 Hz, 6H).

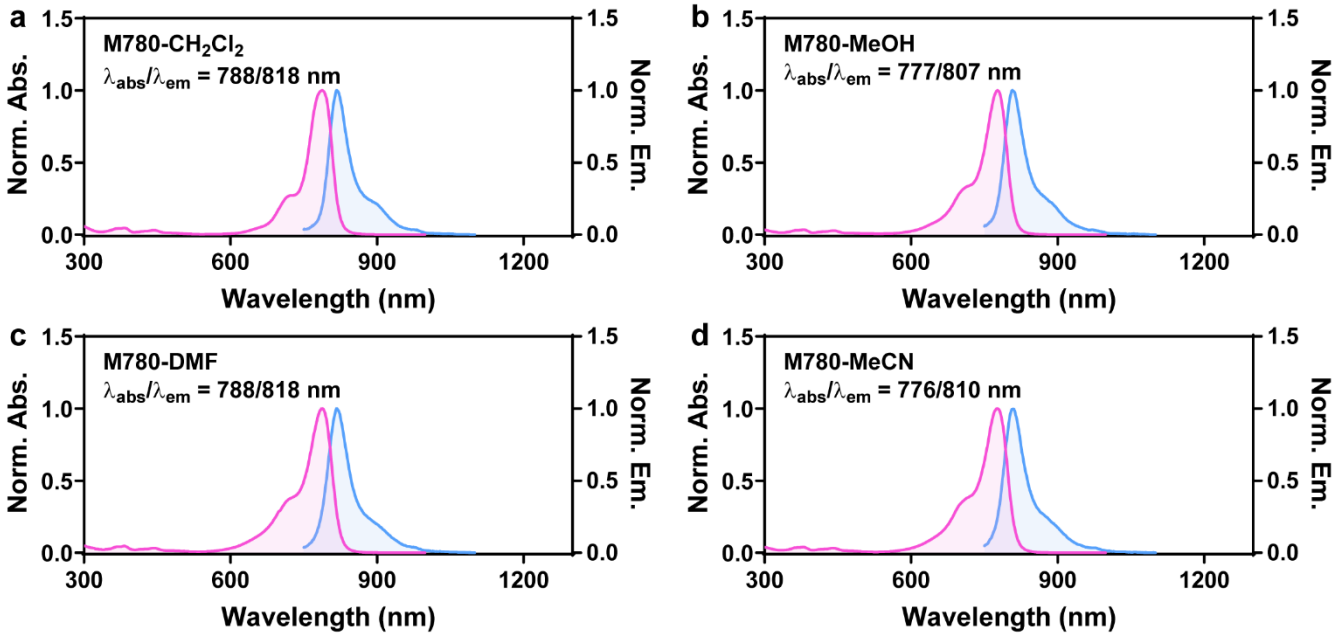

Figure S1: The normalized UV–vis absorption (purple shaded area curve) and fluorescence emission (blue shaded area curve) spectra of **M780** in (a) CH_2Cl_2, (b) MeOH, (c) DMF, (d) MeCN.

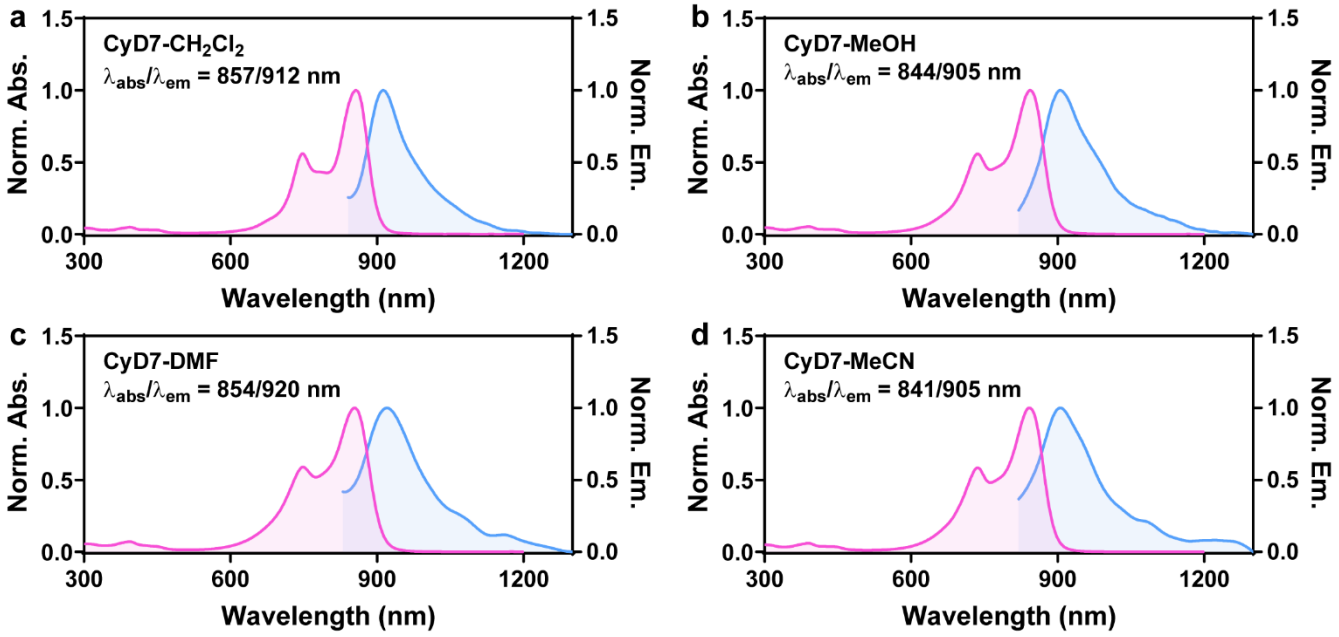

Figure S2: The normalized UV–vis absorption (purple shaded area curve) and fluorescence emission (blue shaded area curve) spectra of **CyD7** in (a) CH_2Cl_2, (b) MeOH, (c) DMF, (d) MeCN.

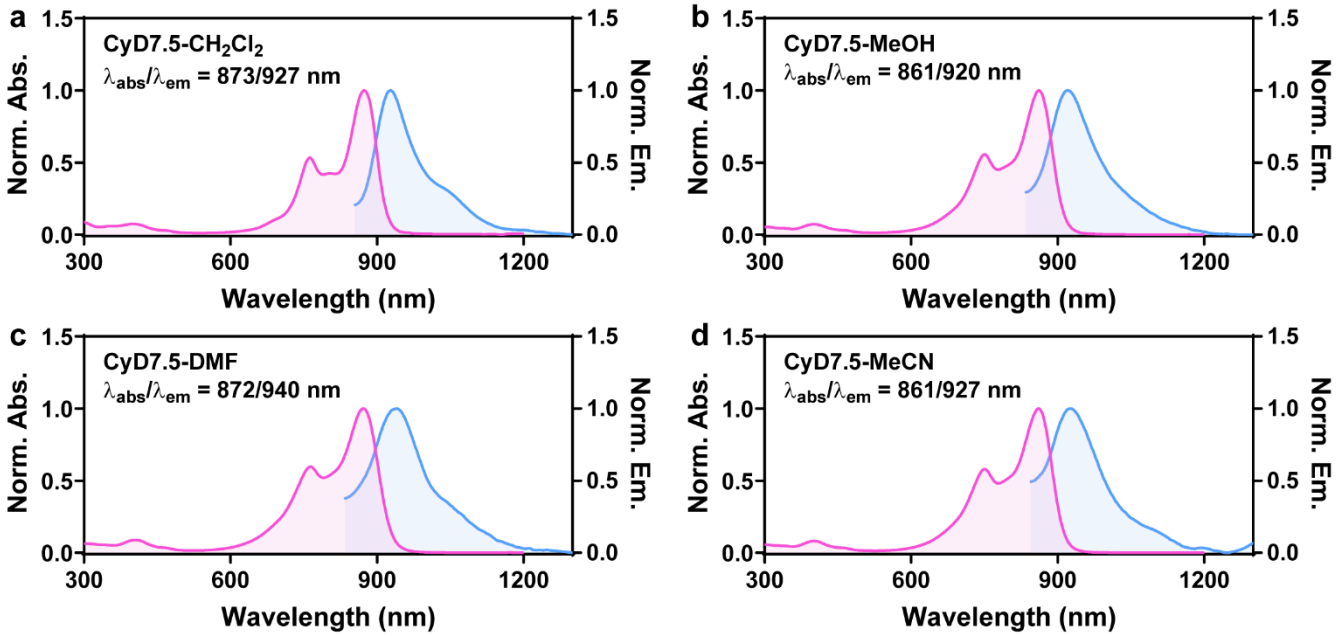

Figure S3: The normalized UV–vis absorption (purple shaded area curve) and fluorescence emission (blue shaded area curve) spectra of **CyD7.5** in (a) CH_2Cl_2, (b) MeOH, (c) DMF, (d) MeCN.

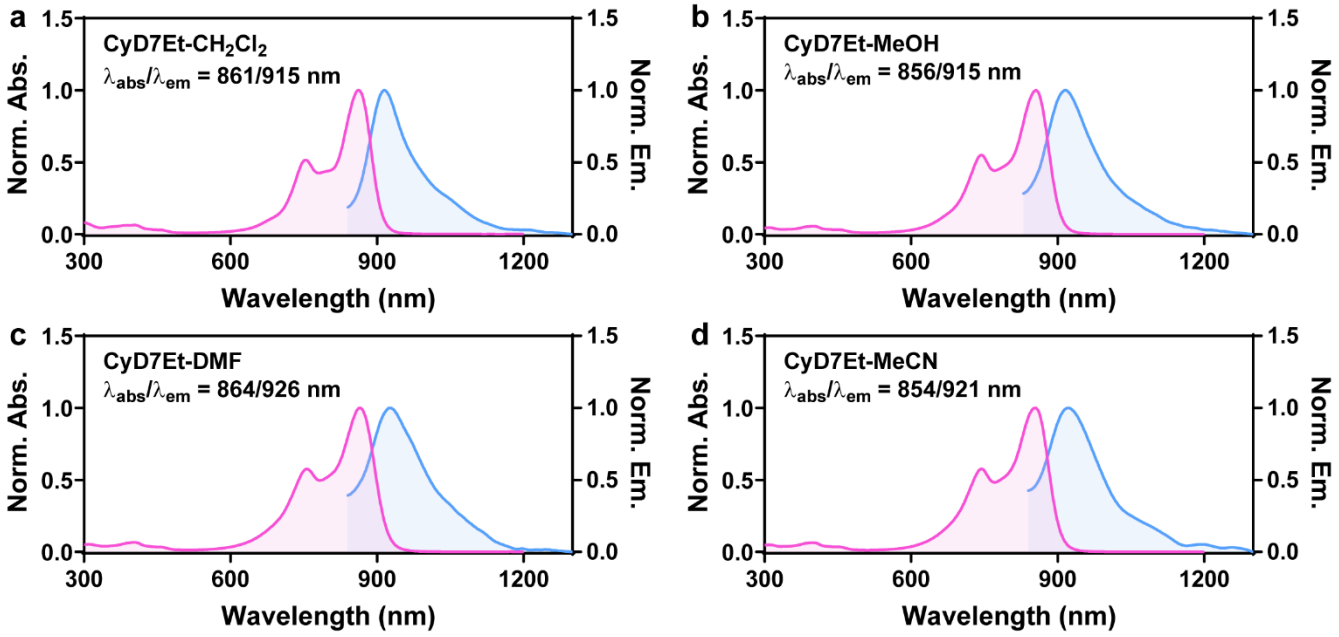

Figure S4: The normalized UV–vis absorption (purple shaded area curve) and fluorescence emission (blue shaded area curve) spectra of **CyD7Et** in (a) CH_2Cl_2, (b) MeOH, (c) DMF, (d) MeCN.

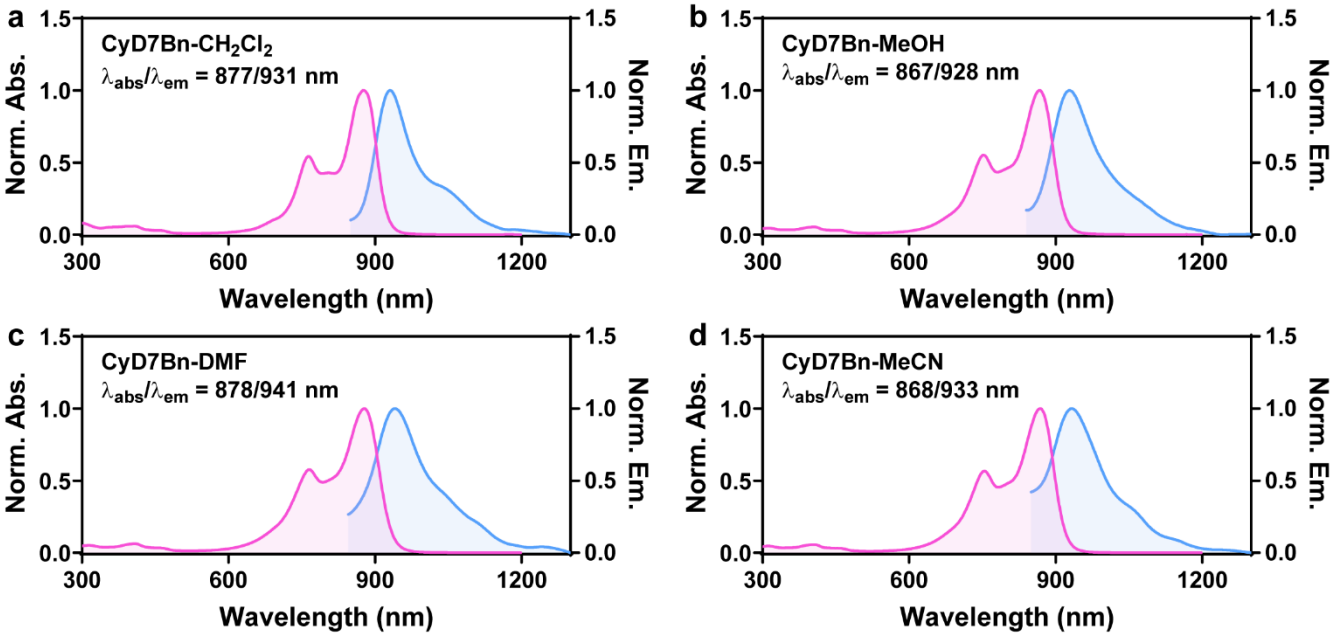

Figure S5: The normalized UV–vis absorption (purple shaded area curve) and fluorescence emission (blue shaded area curve) spectra of **CyD7Bn** in (a) CH_2Cl_2, (b) MeOH, (c) DMF, (d) MeCN.

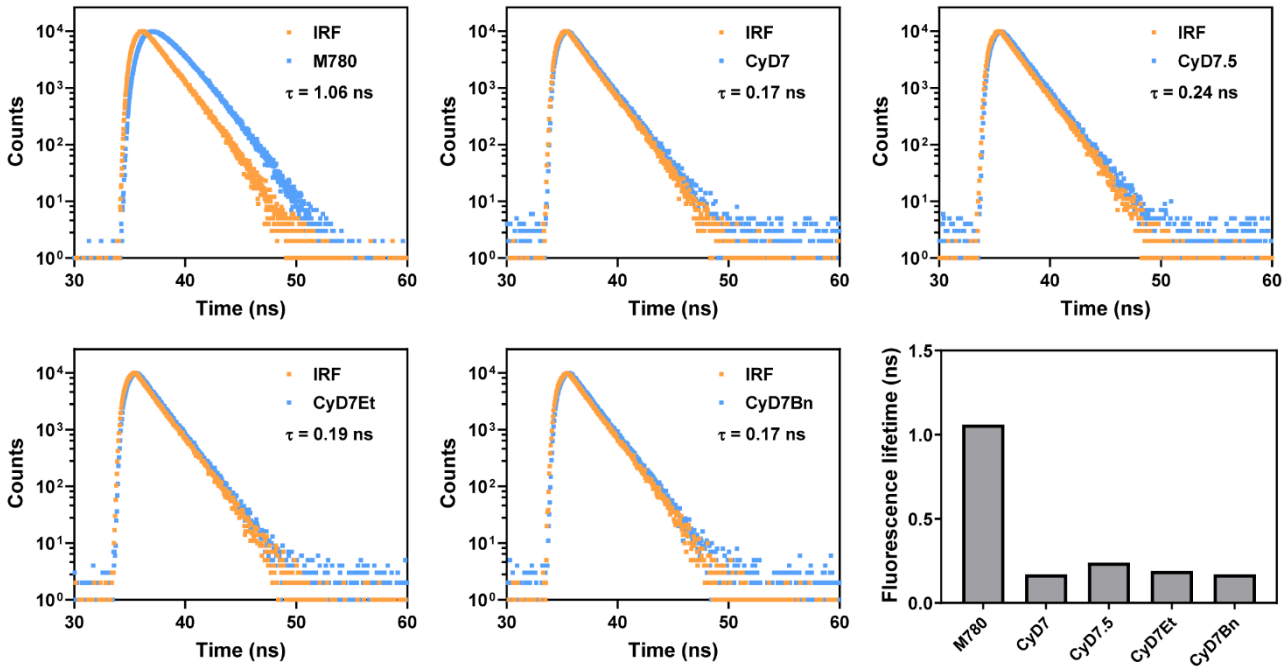

Figure S6: Fluorescence lifetime spectra of dyes in CH_2Cl_2.

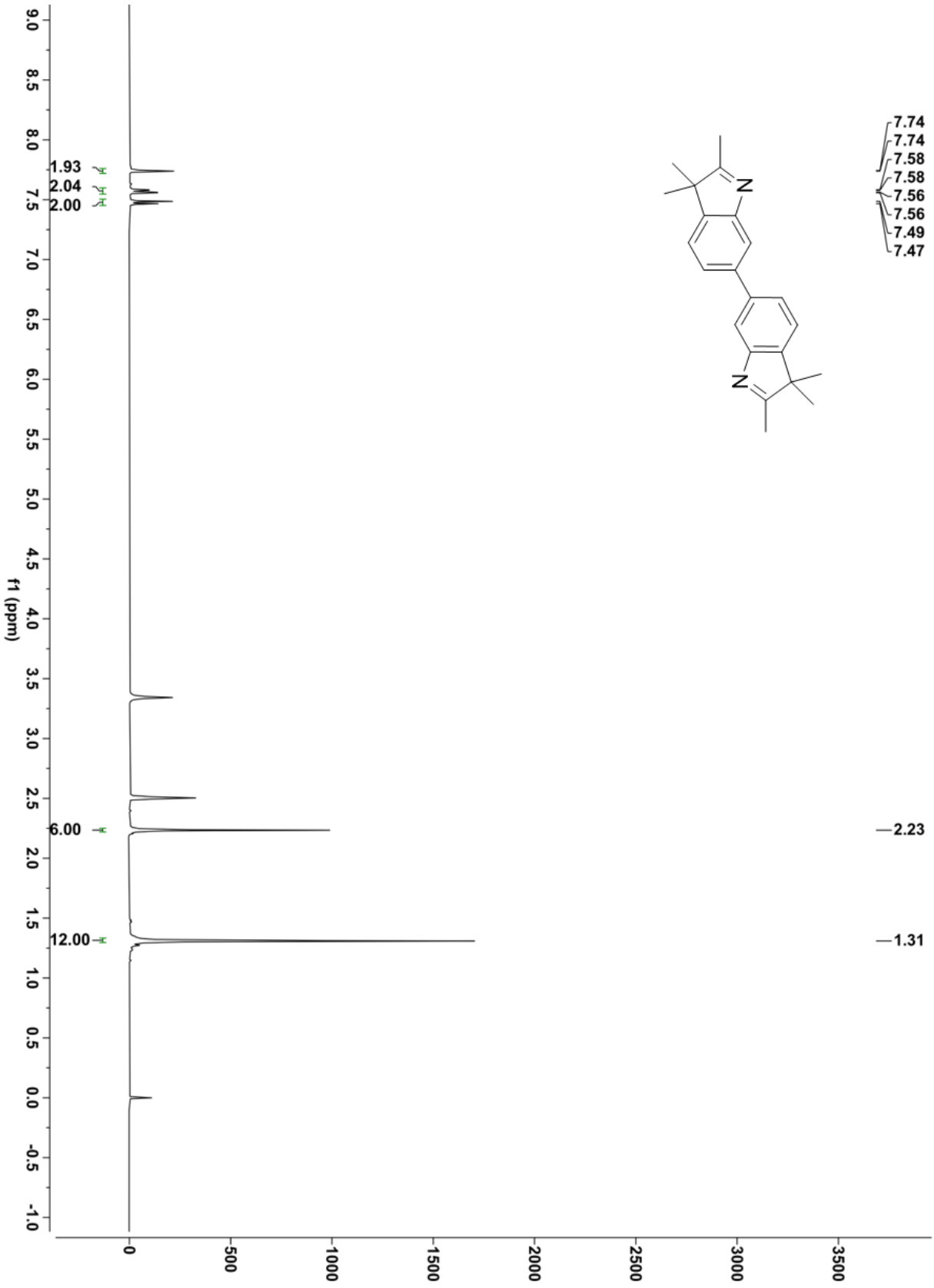

Figure S7: ^{1}H NMR spectrum of **3** in DMSO-d_6 (400 MHz).

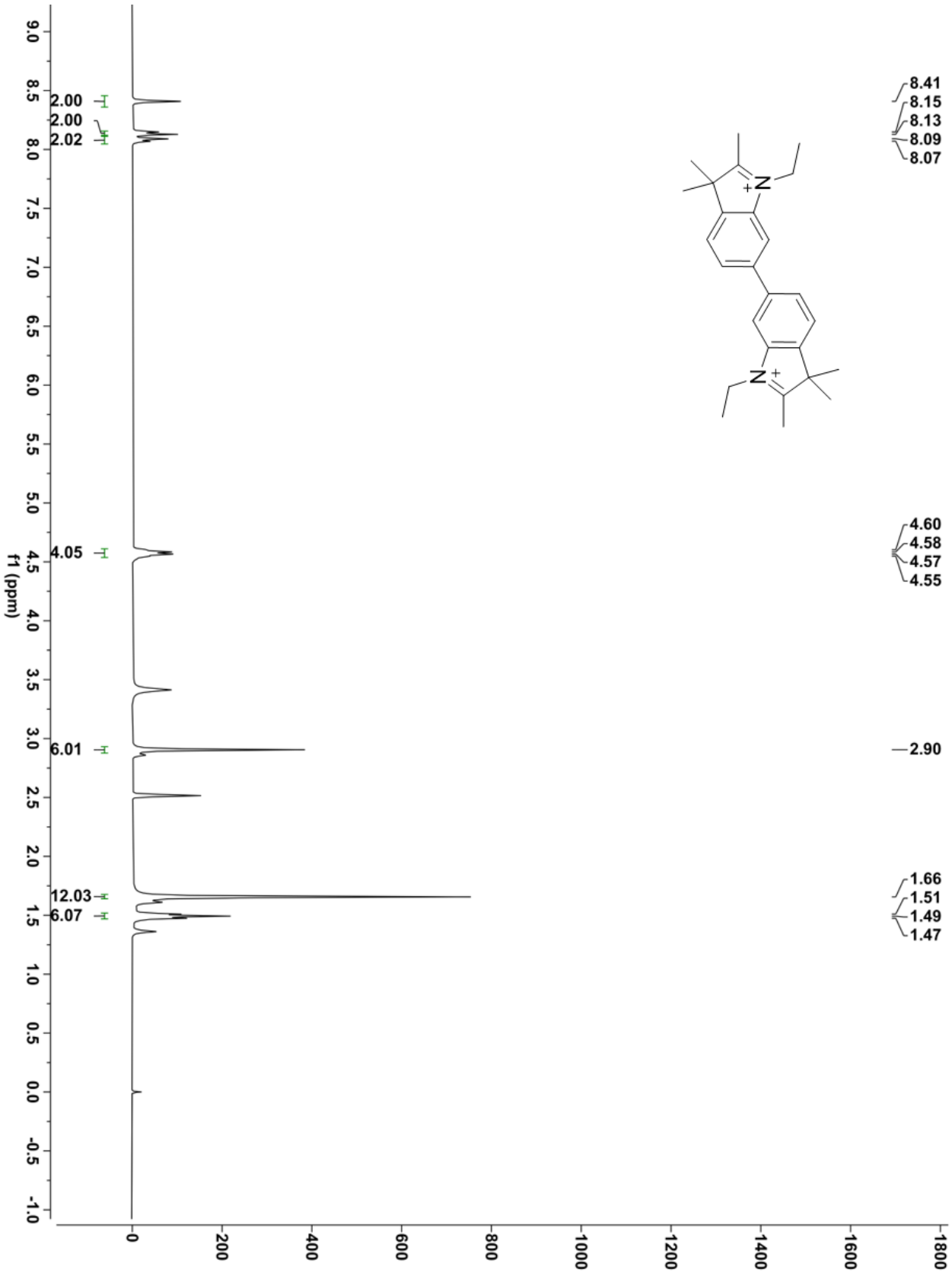

Figure S8: ^{1}H NMR spectrum of **M1** in DMSO-d_6 (400 MHz).

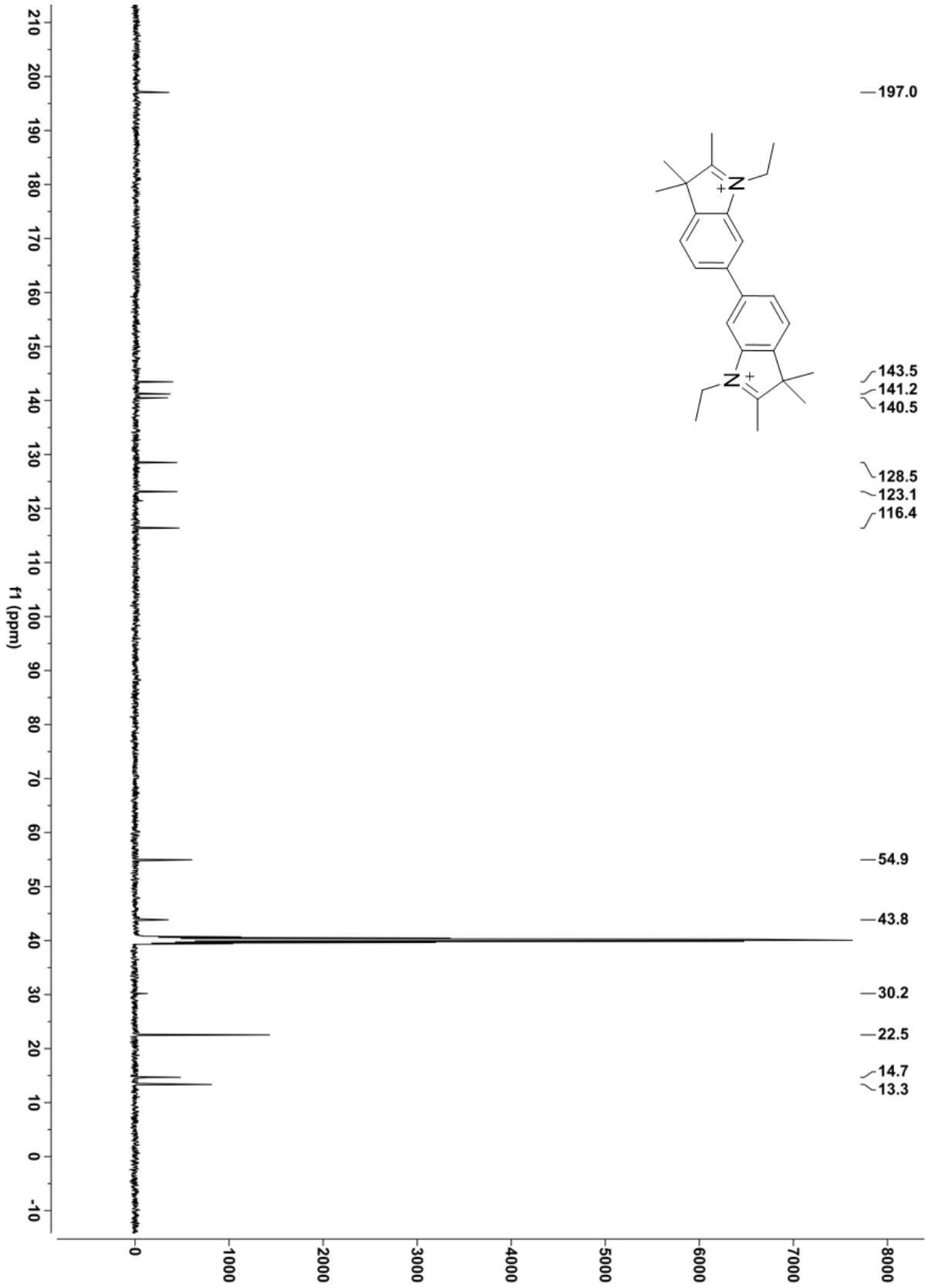

Figure S9: ^{13}C NMR spectrum of **M1** in DMSO-d_6 (100 MHz).

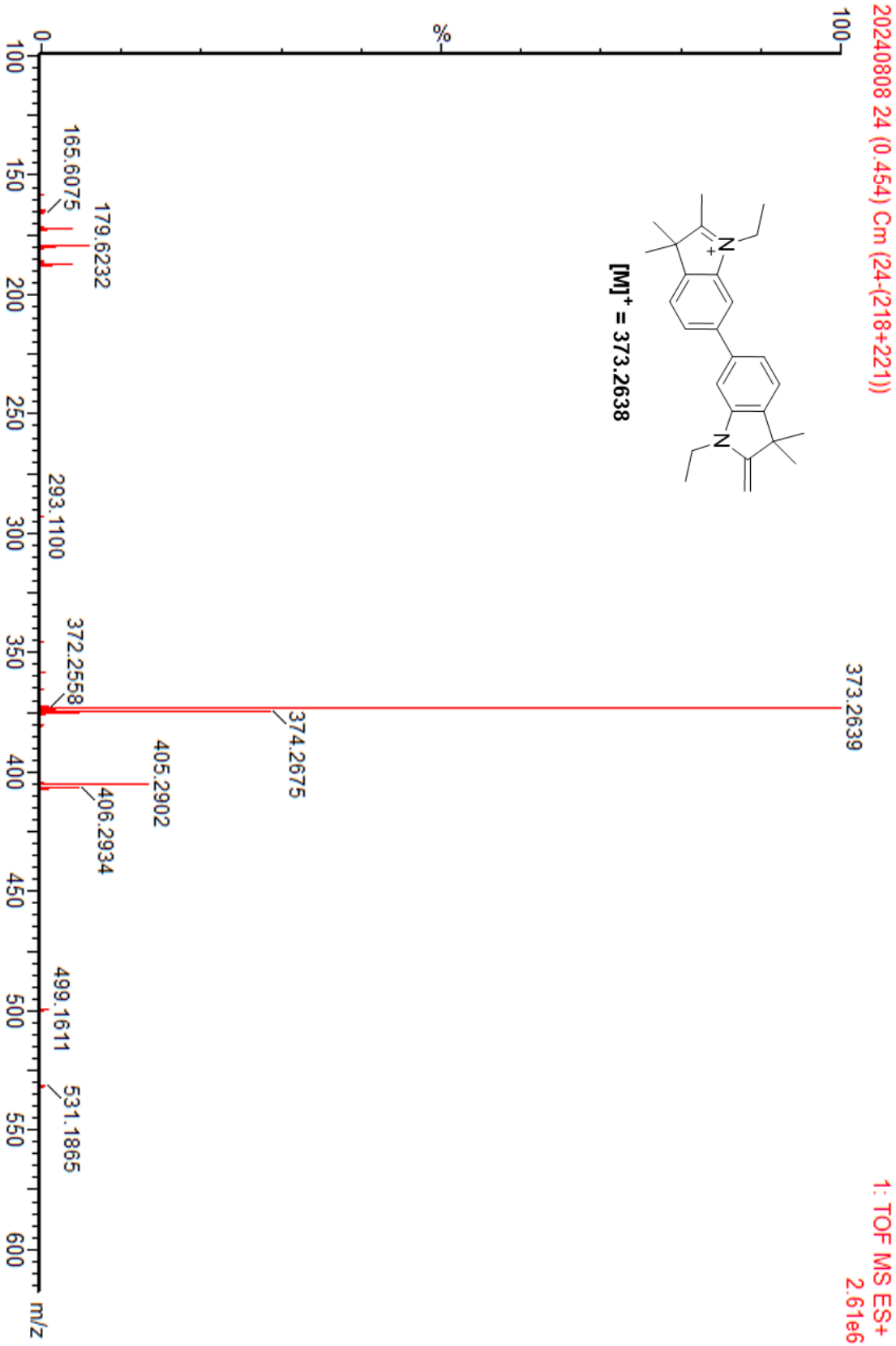

Figure S10: HRMS-ESI spectrum of **M1**.

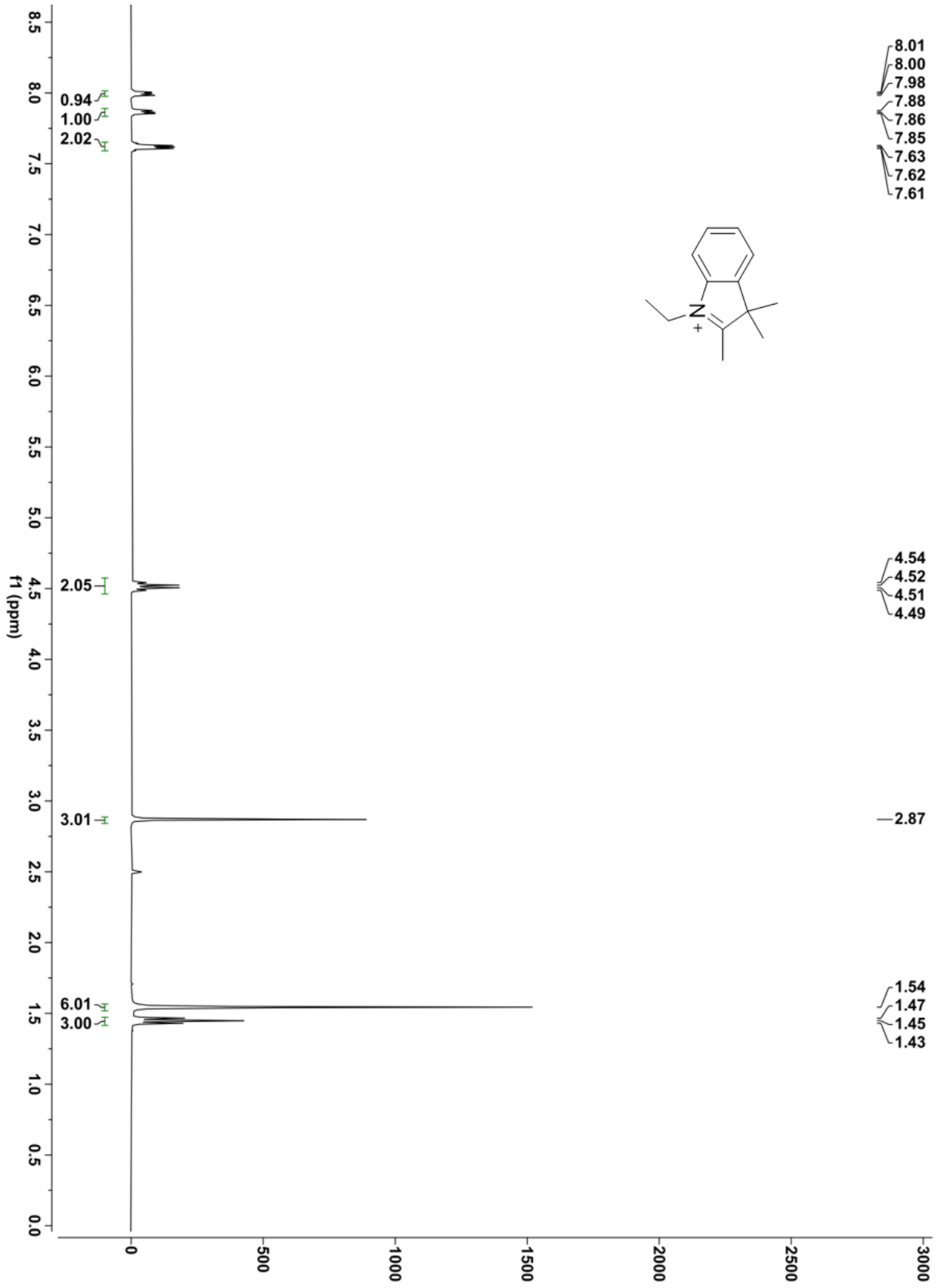

Figure S11: ^{1}H NMR spectrum of **M2** in DMSO-d_6 (400 MHz).

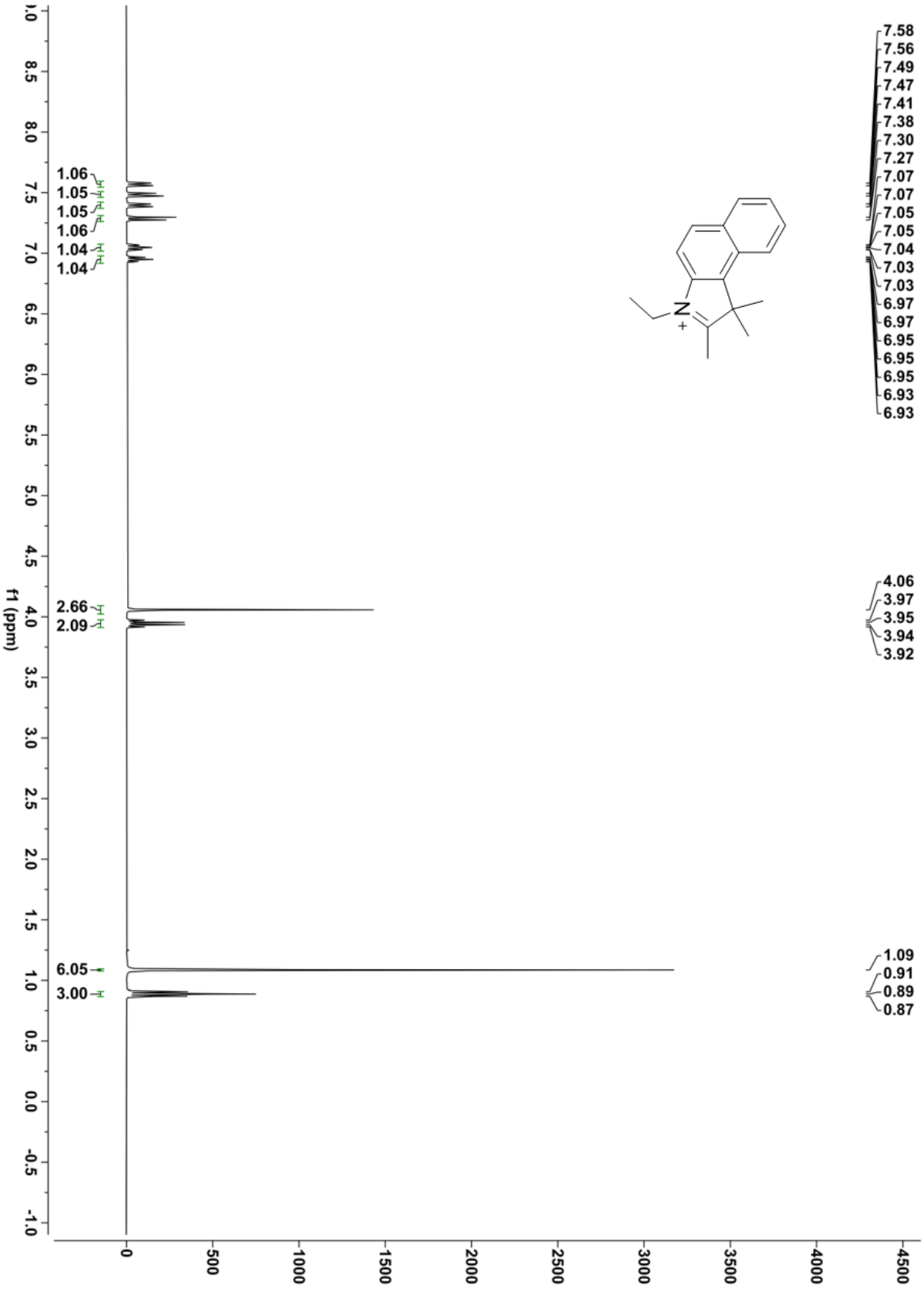

Figure S12: ^{1}H NMR spectrum of **M3** in DMSO-d_6 (400 MHz).

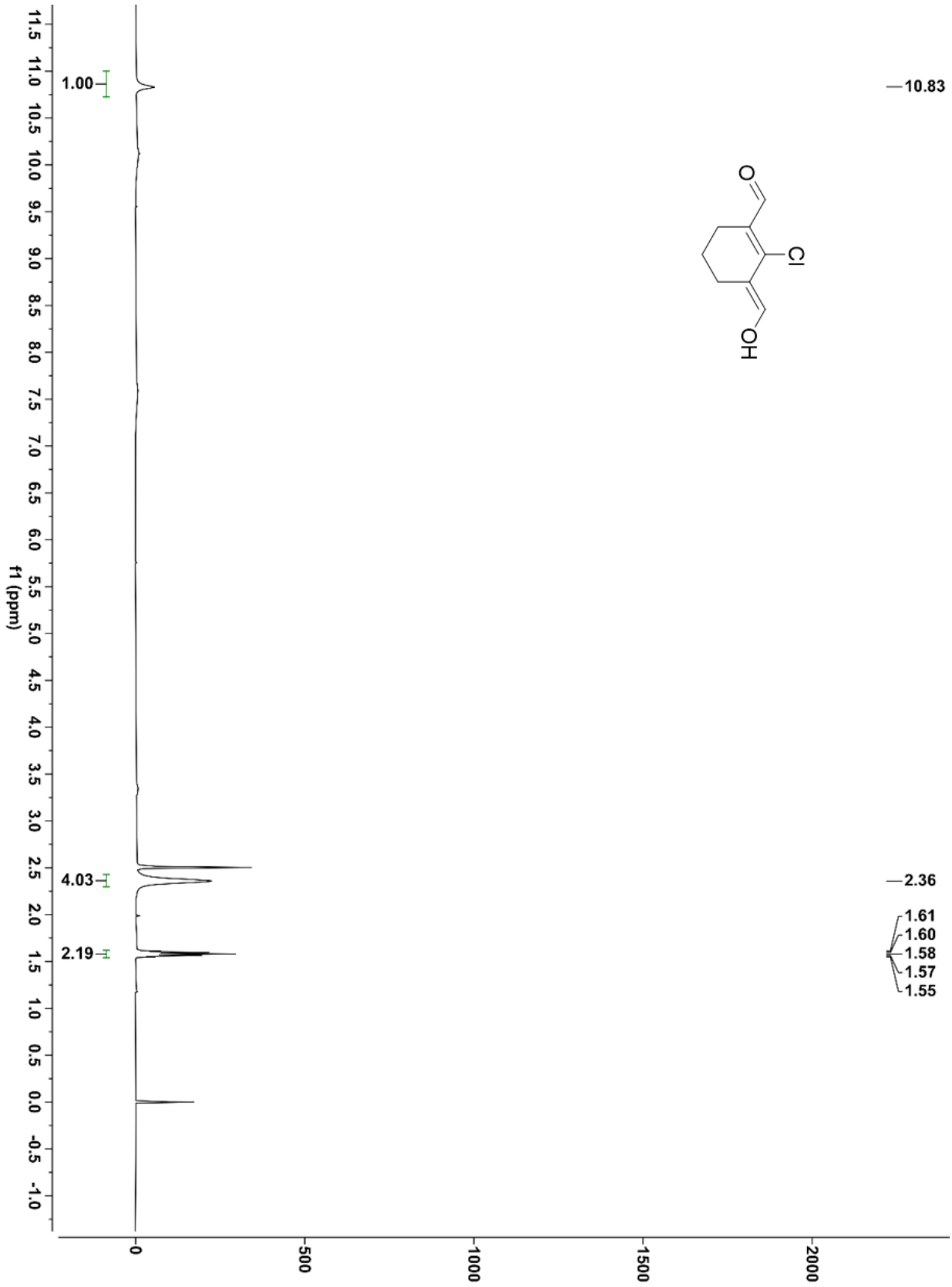

Figure S13: ^{1}H NMR spectrum of **N1** in DMSO-d_6 (400 MHz).

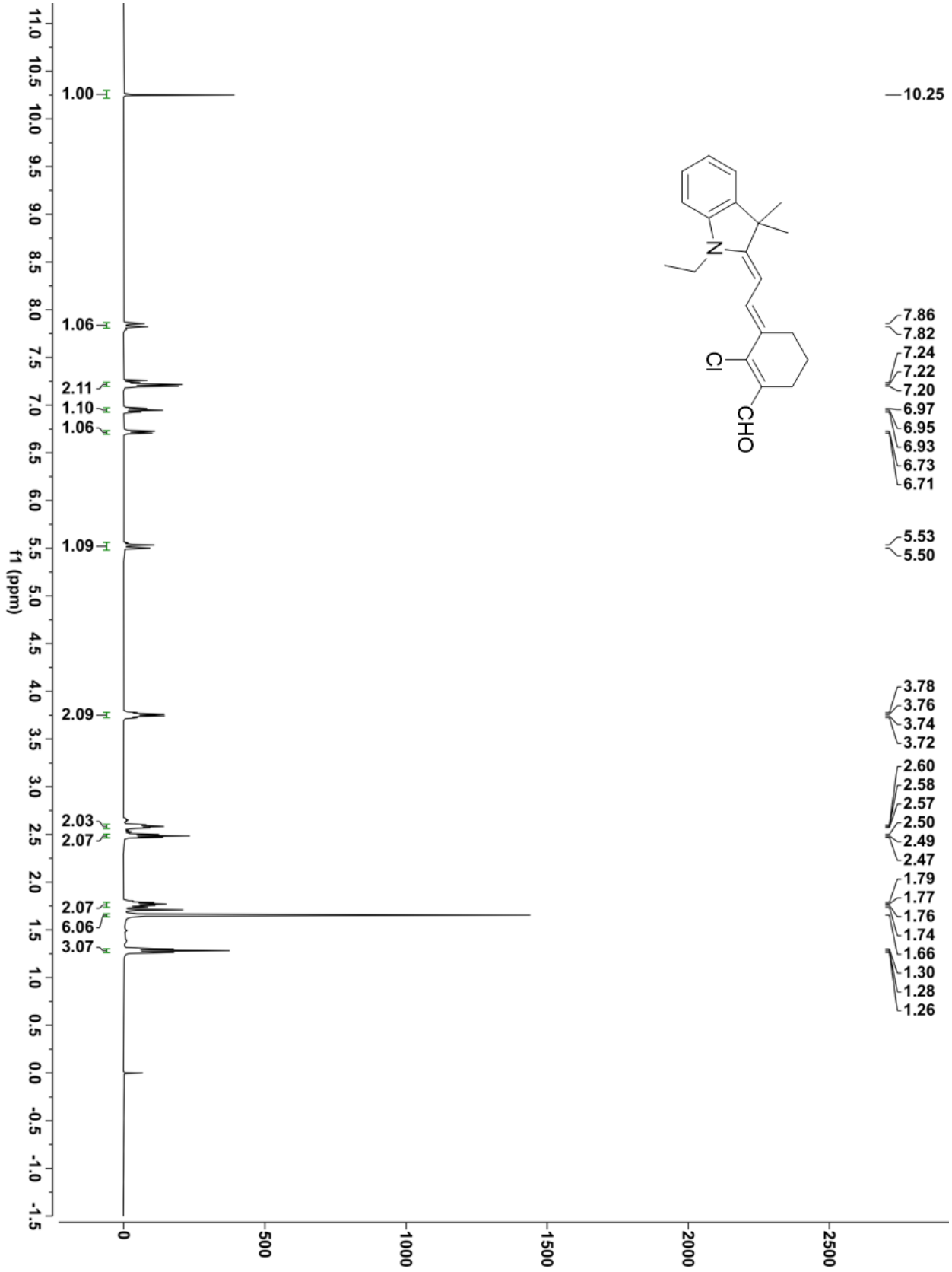

Figure S14: ^{1}H NMR spectrum of **W1** in $CDCl_3$ (400 MHz).

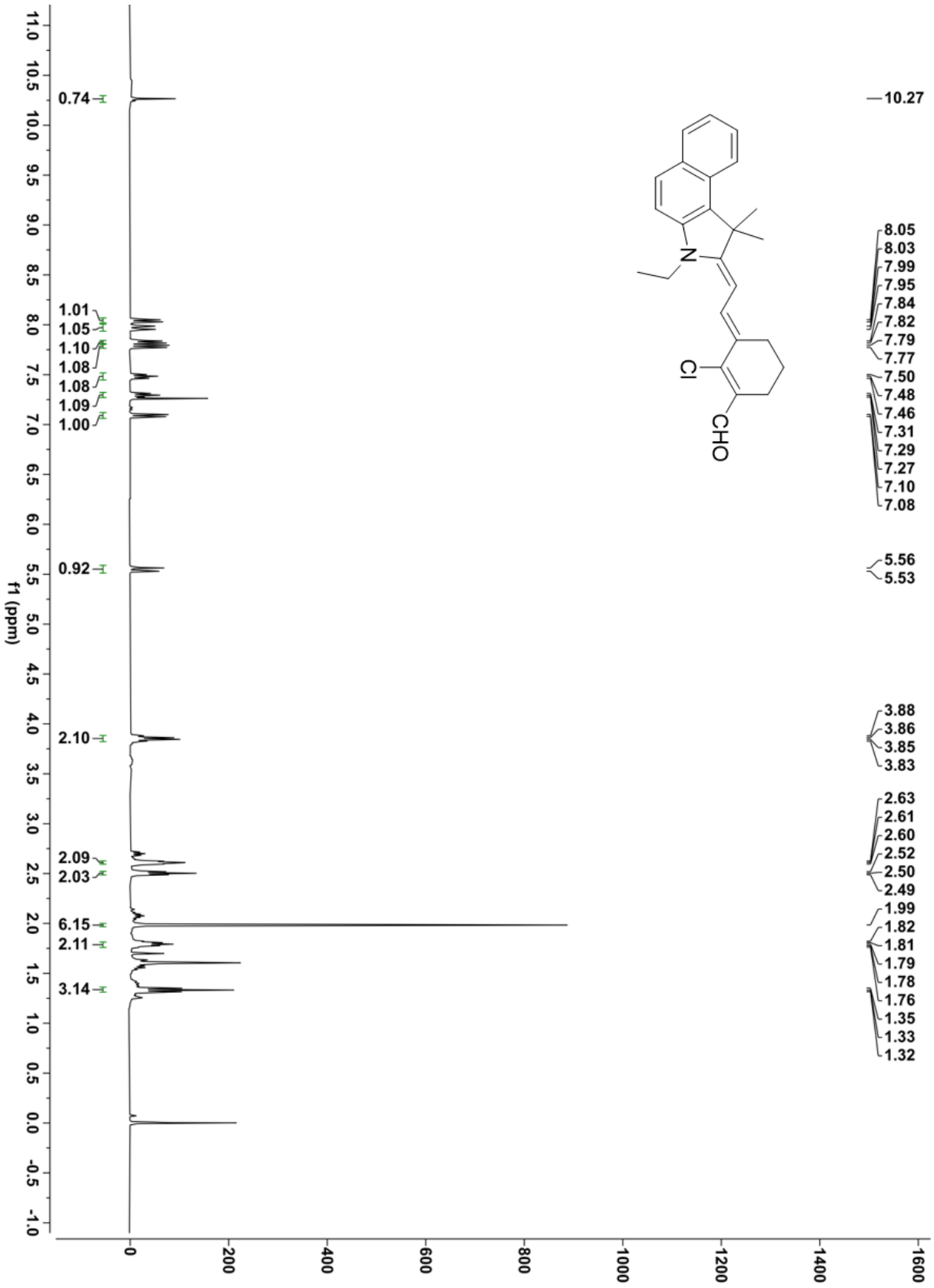

Figure S15: ^{1}H NMR spectrum of **W2** in $CDCl_3$ (400 MHz).

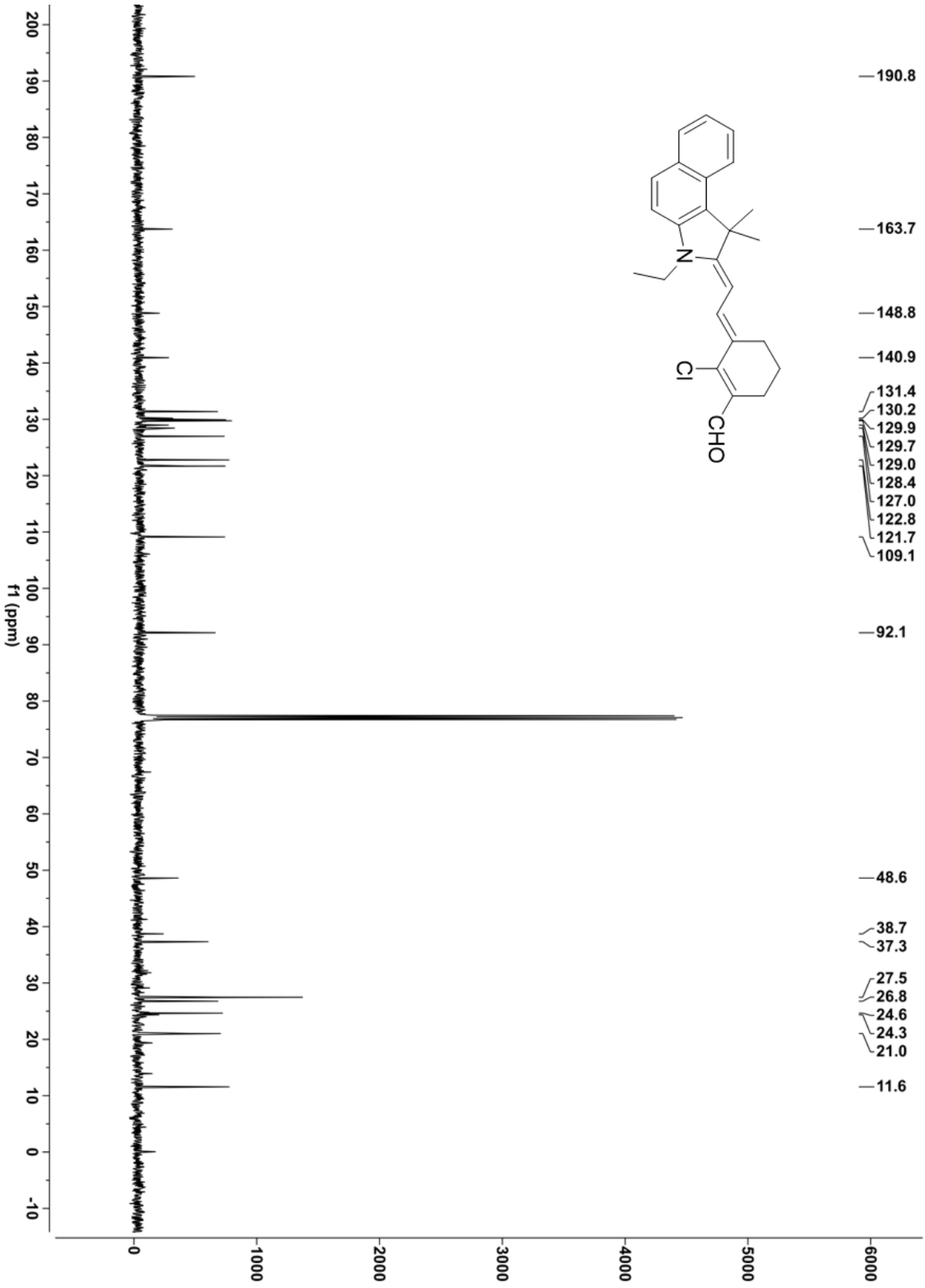

Figure S16: ^{13}C NMR spectrum of **W2** in $CDCl_3$ (100 MHz).

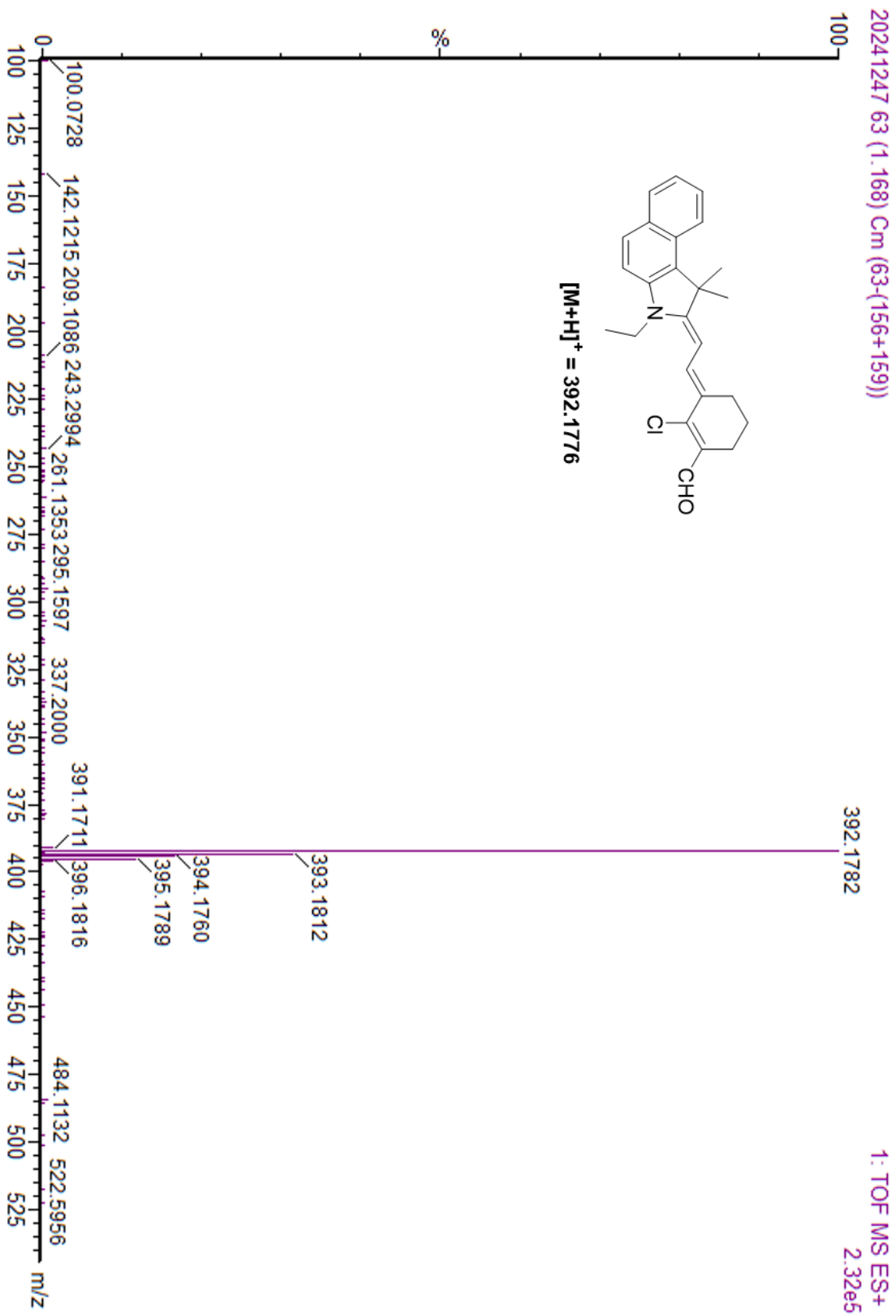

Figure S17: HRMS-ESI spectrum of **W2**.

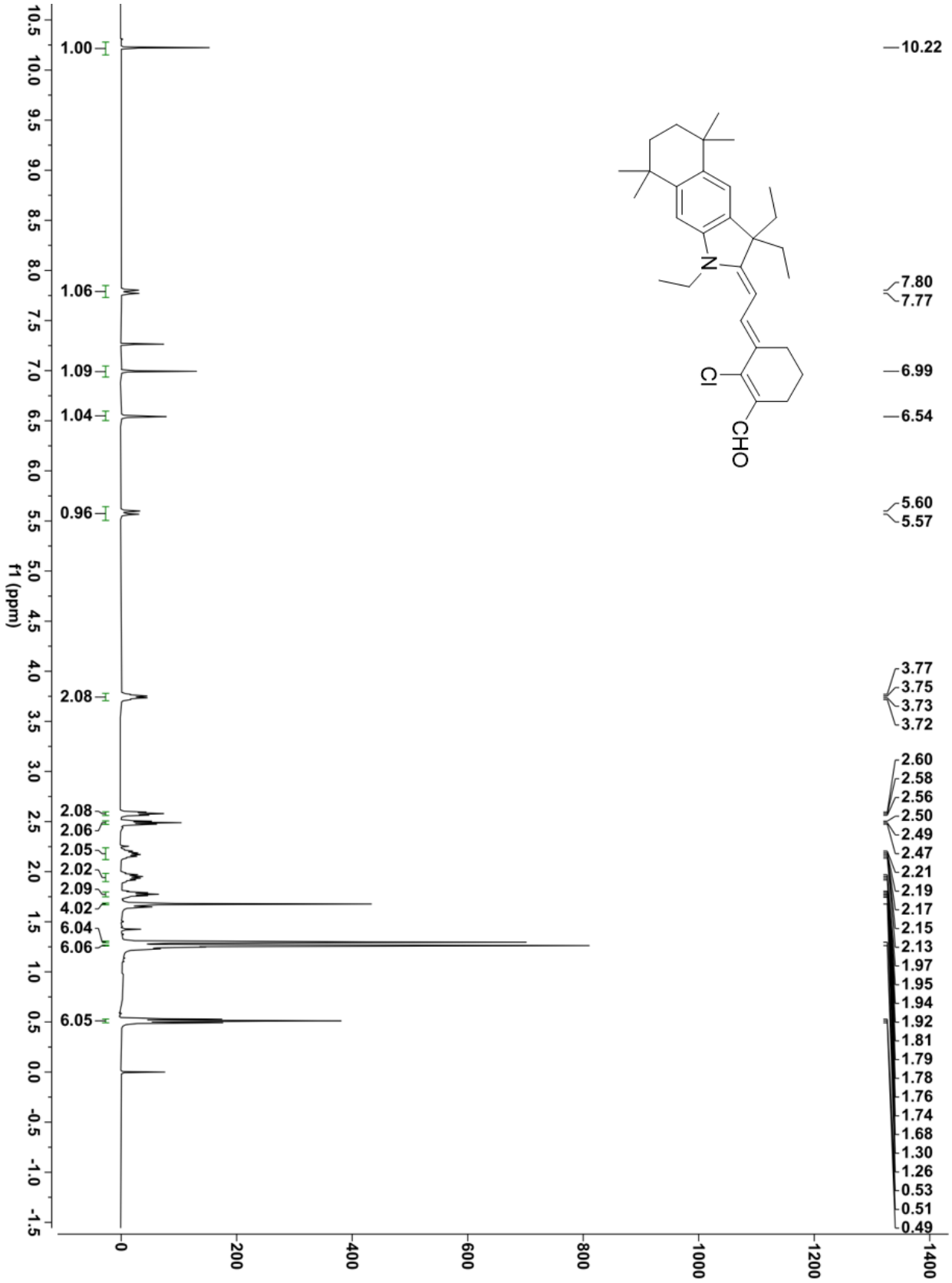

Figure S18: ^{1}H NMR spectrum of **W3** in $CDCl_3$ (400 MHz).

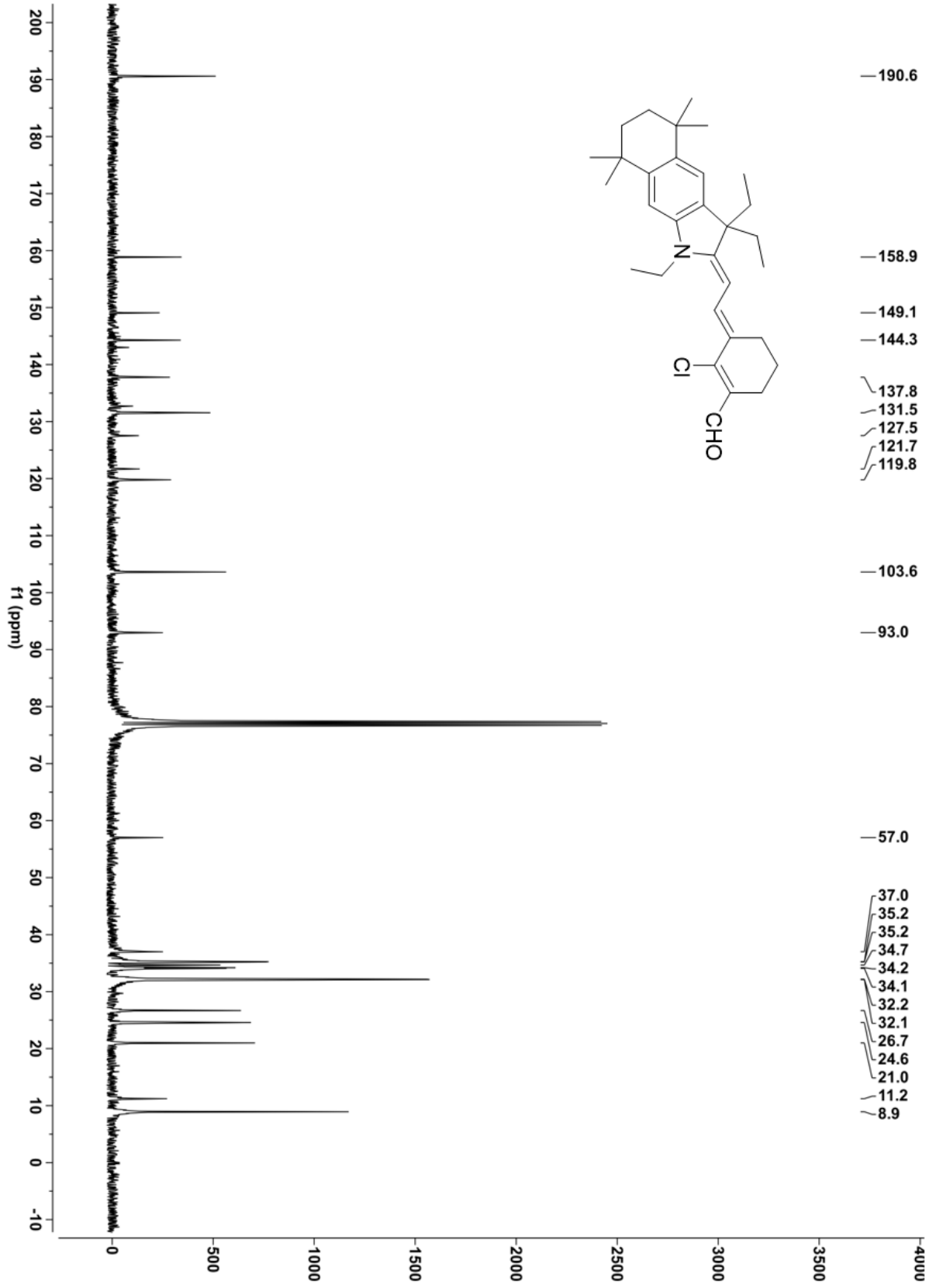

Figure S19: ^{13}C NMR spectrum of **W3** in $CDCl_3$ (100 MHz).

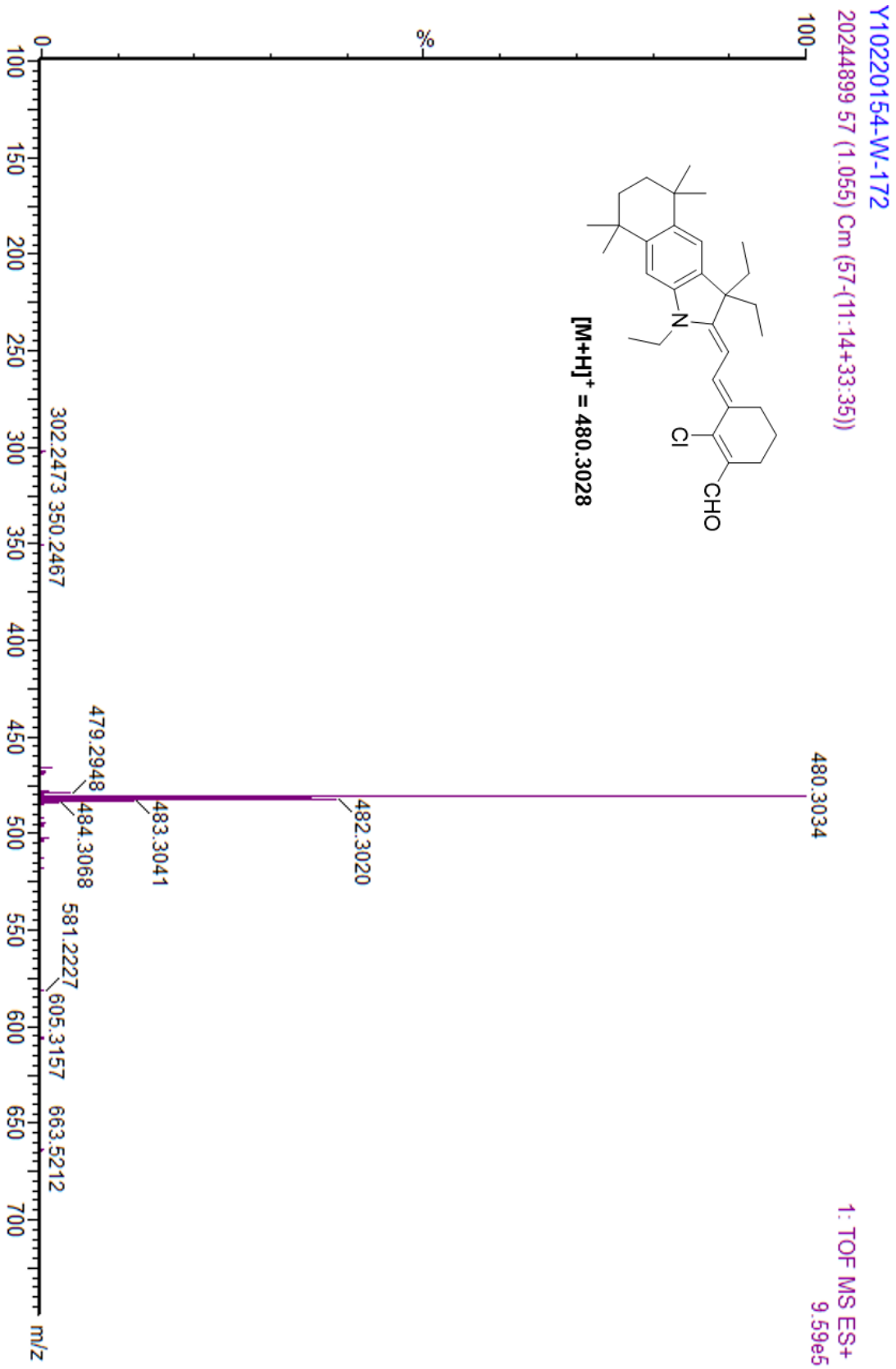

Figure S20: HRMS-ESI spectrum of **M3**.

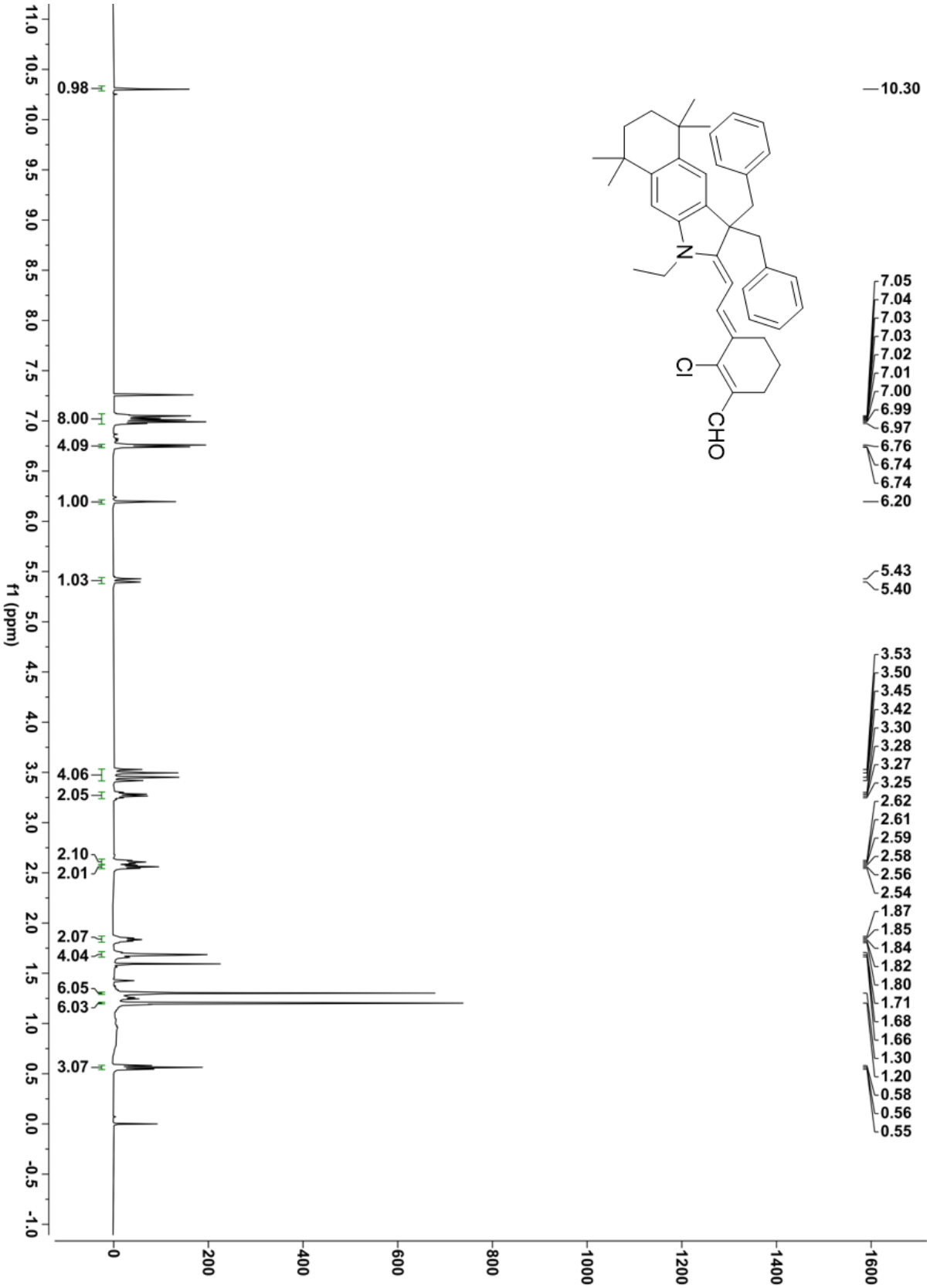

Figure S21: ^{1}H NMR spectrum of **W4** in $CDCl_3$ (400 MHz).

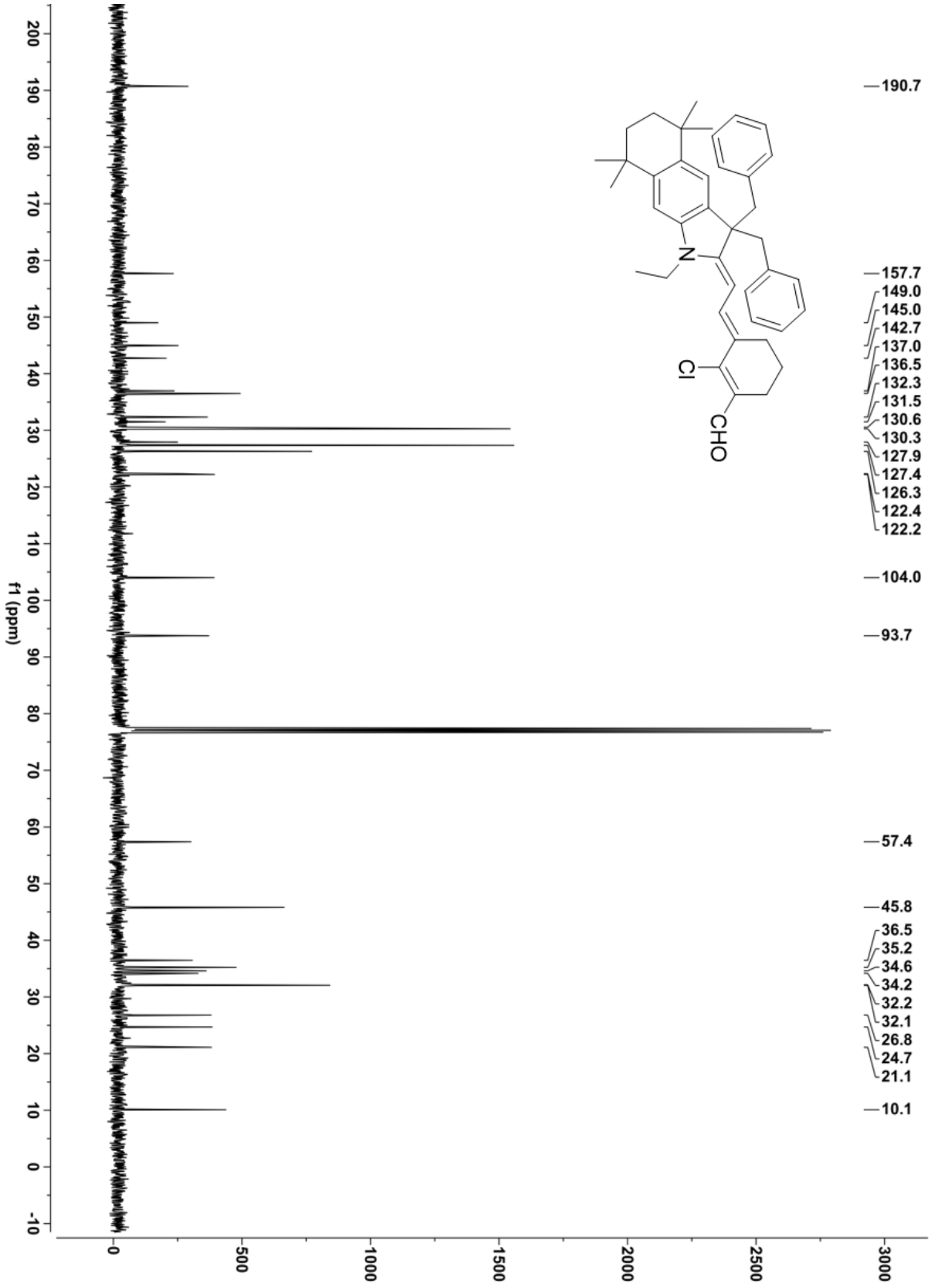

Figure S22: ^{13}C NMR spectrum of **W4** in $CDCl_3$ (100 MHz).

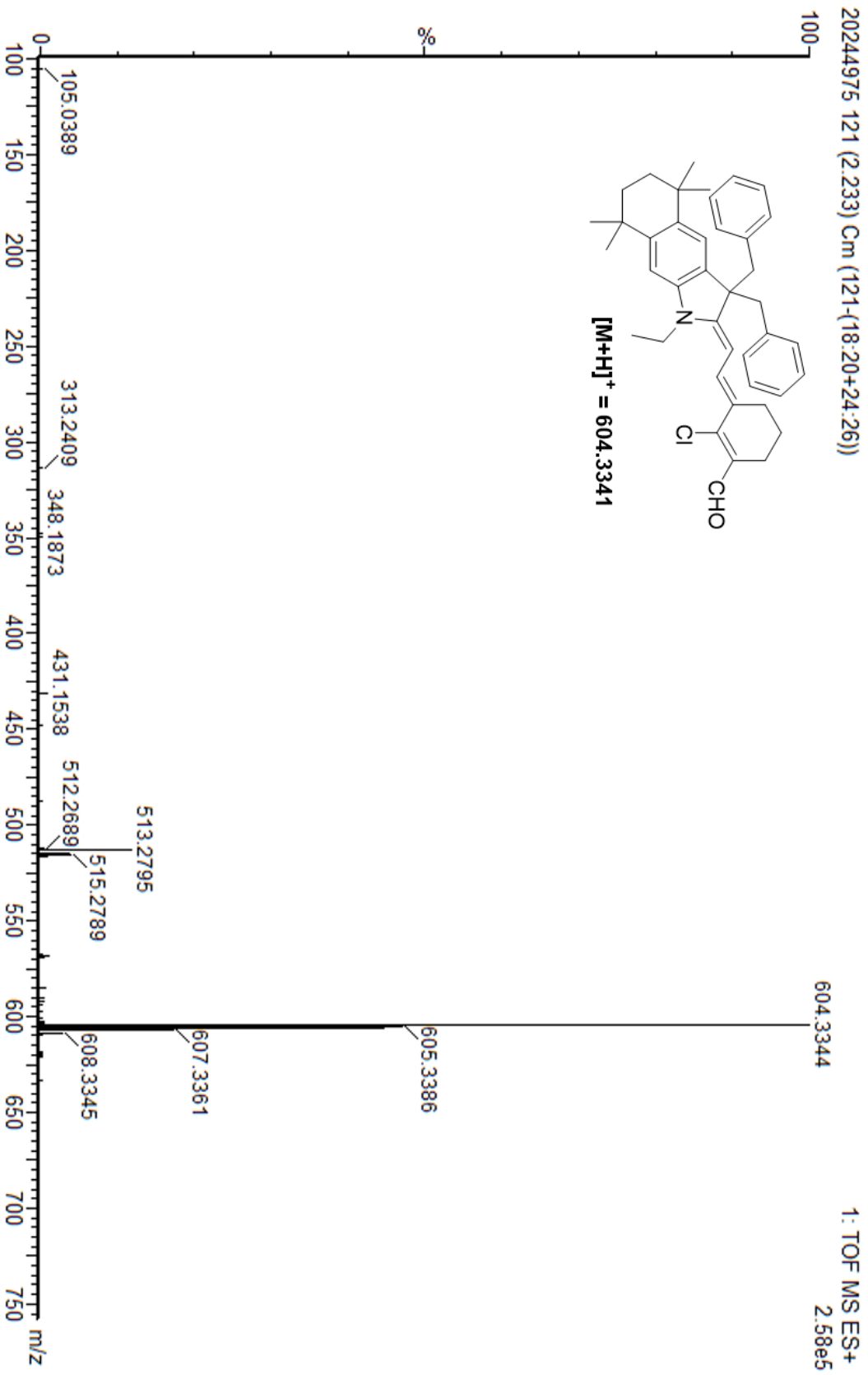

Figure S23: HRMS-ESI spectrum of **M4**.

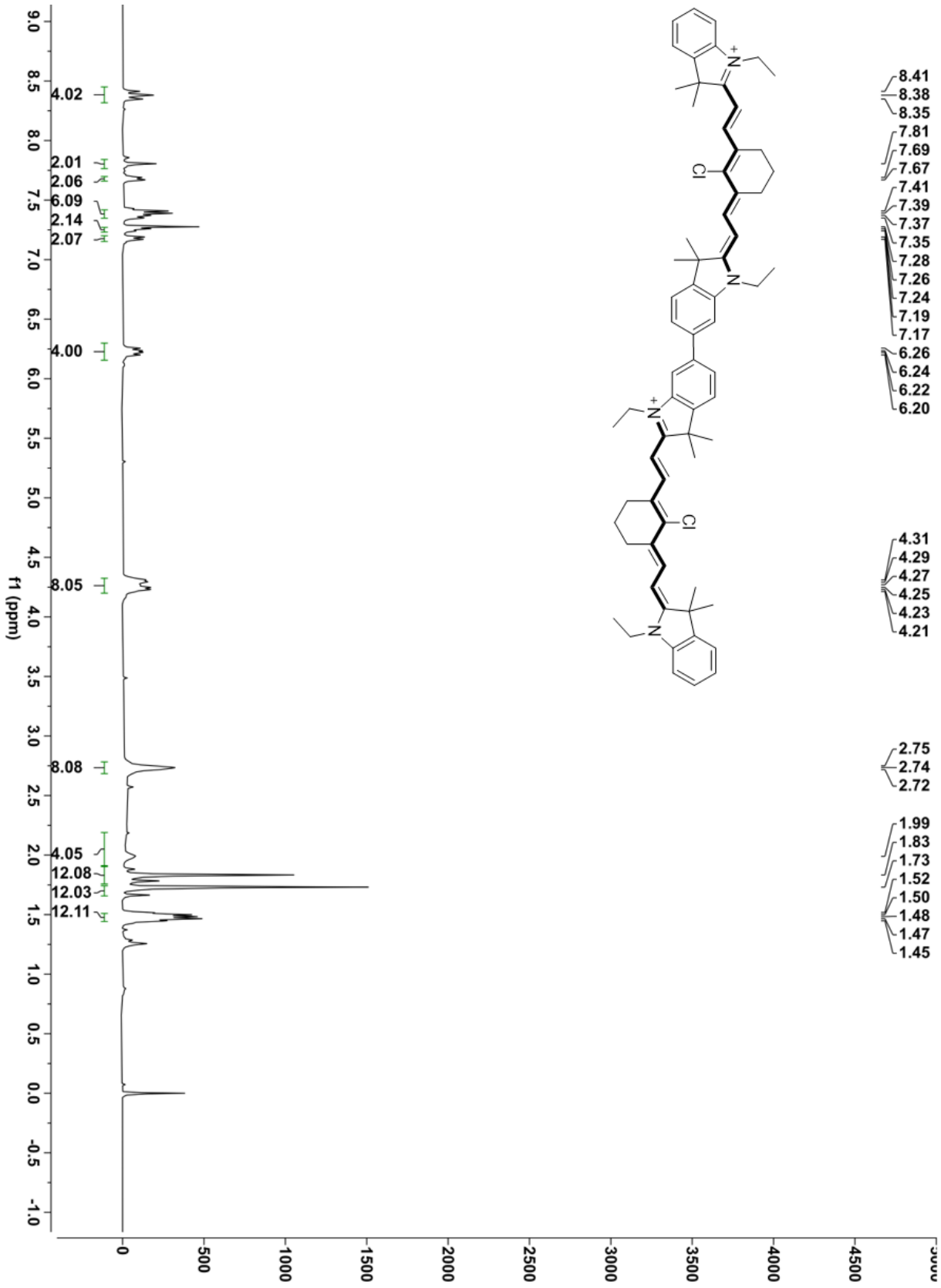

Figure S24: ^{1}H NMR spectrum of **CyD7** in $CDCl_3$ (400 MHz).

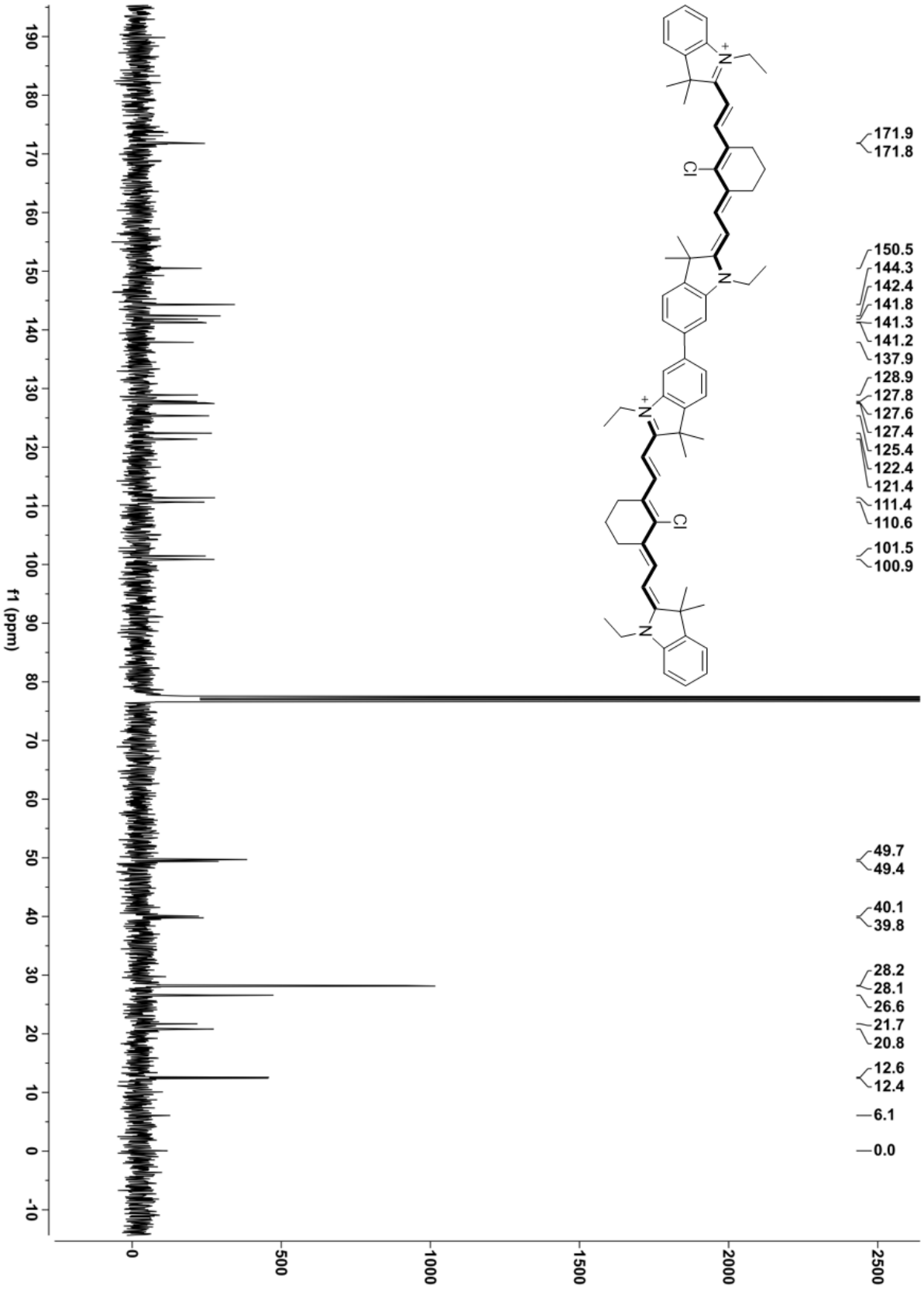

Figure S25: ^{13}C NMR spectrum of **CyD7** in $CDCl_3$ (100 MHz).

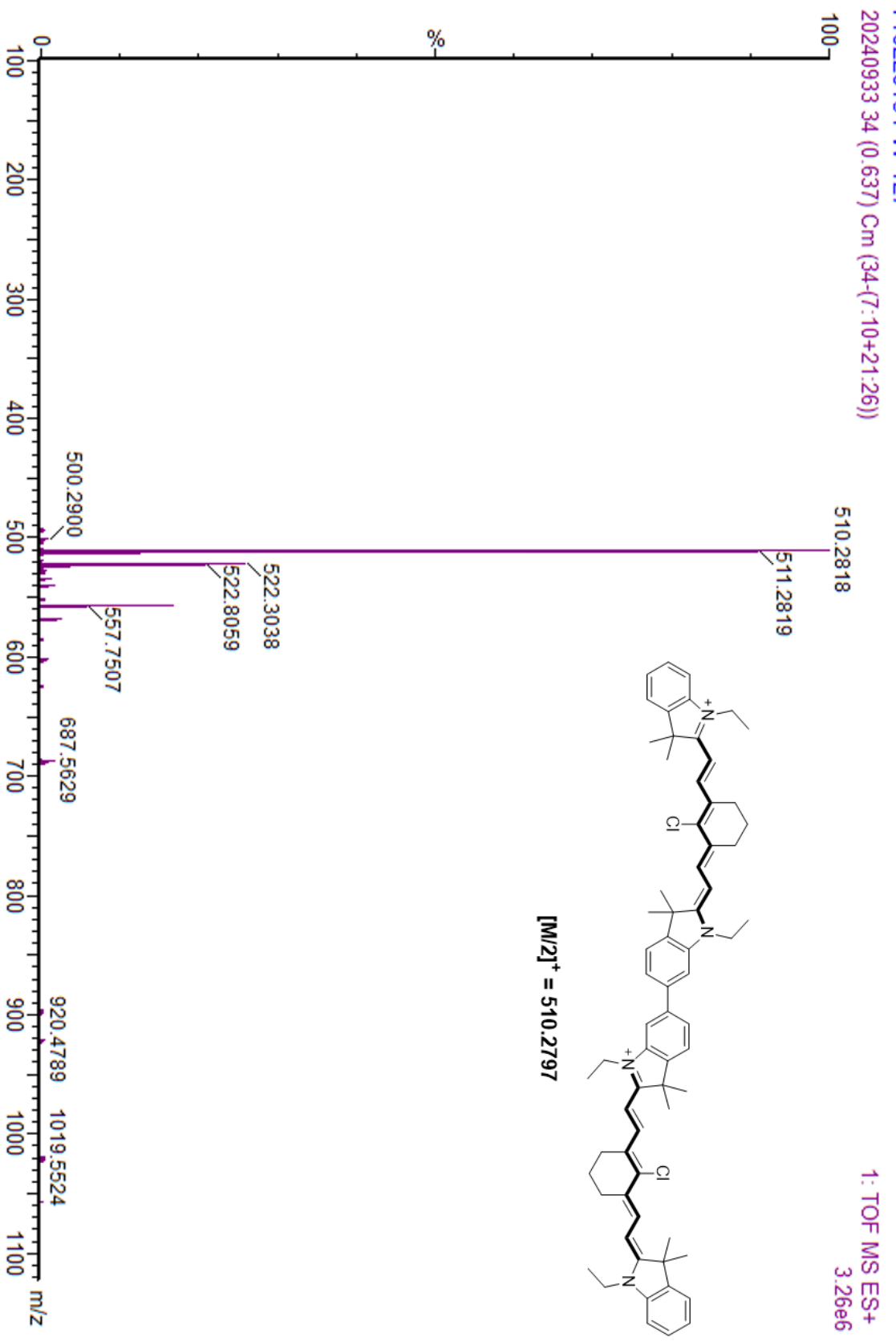

Figure S26: HRMS-ESI spectrum of **CyD7**.

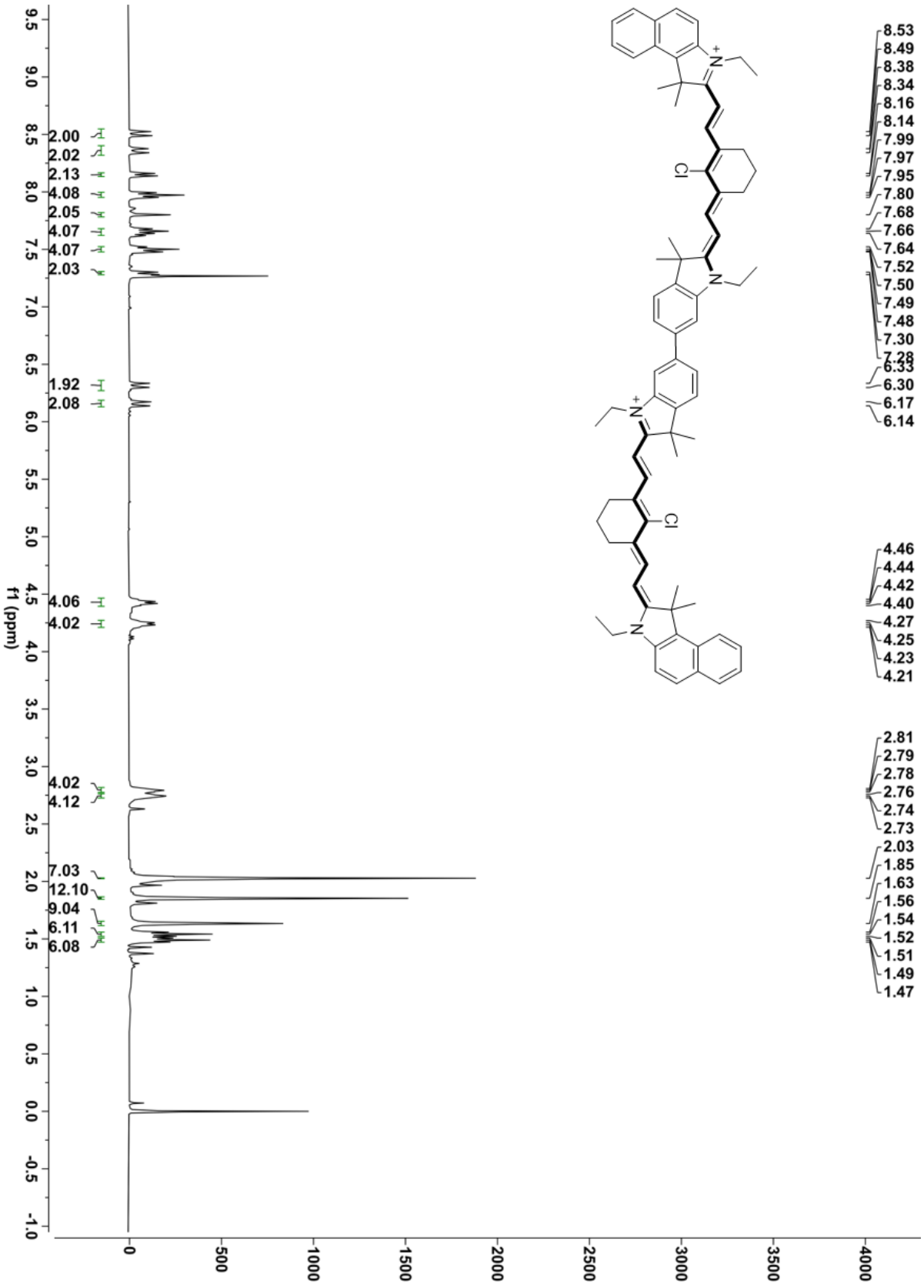

Figure S27: ^{1}H NMR spectrum of **CyD7.5** in $CDCl_3$ (400 MHz).

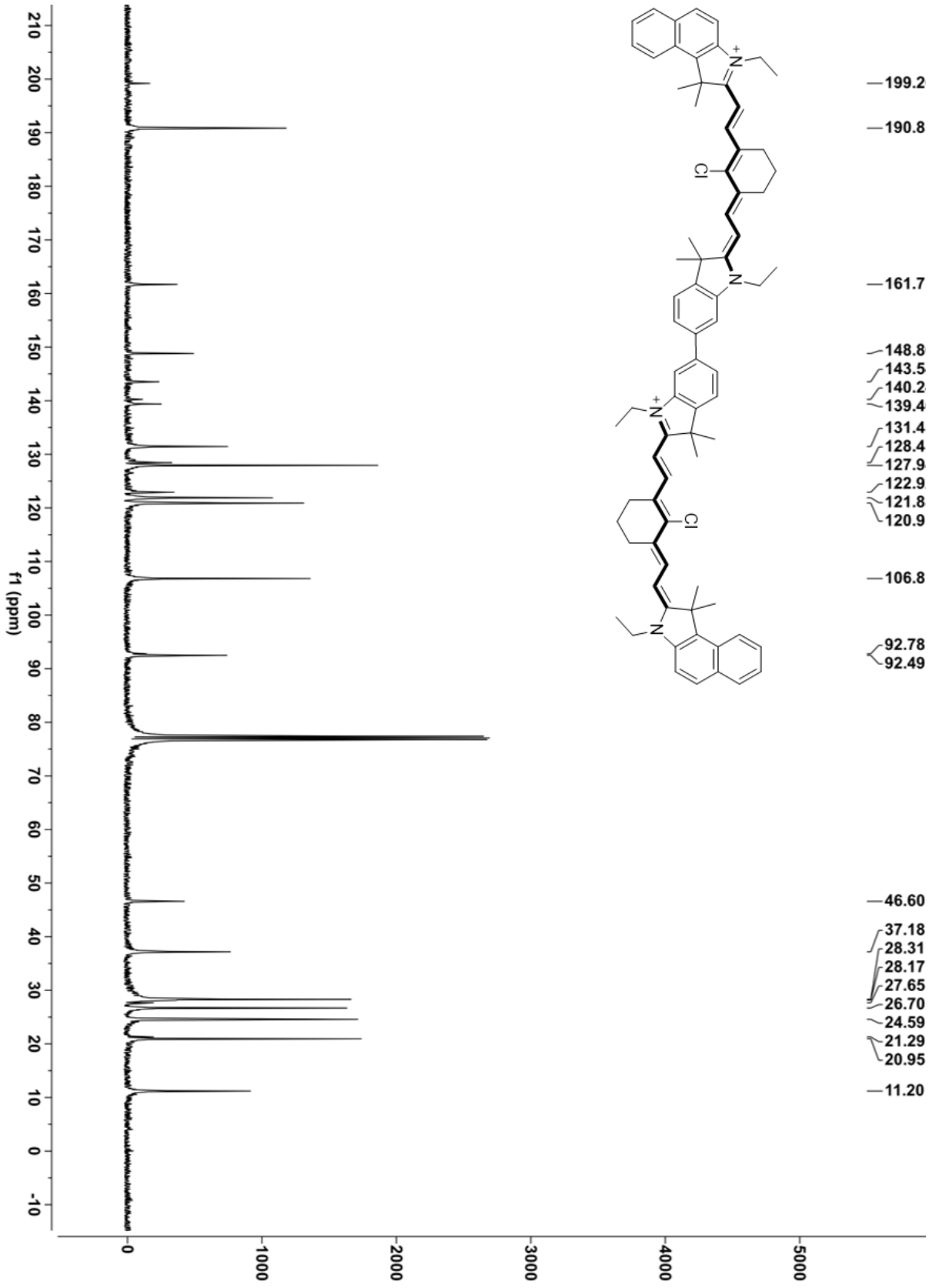

Figure S28: ^{13}C NMR spectrum of **CyD7.5** in $CDCl_3$ (100 MHz).

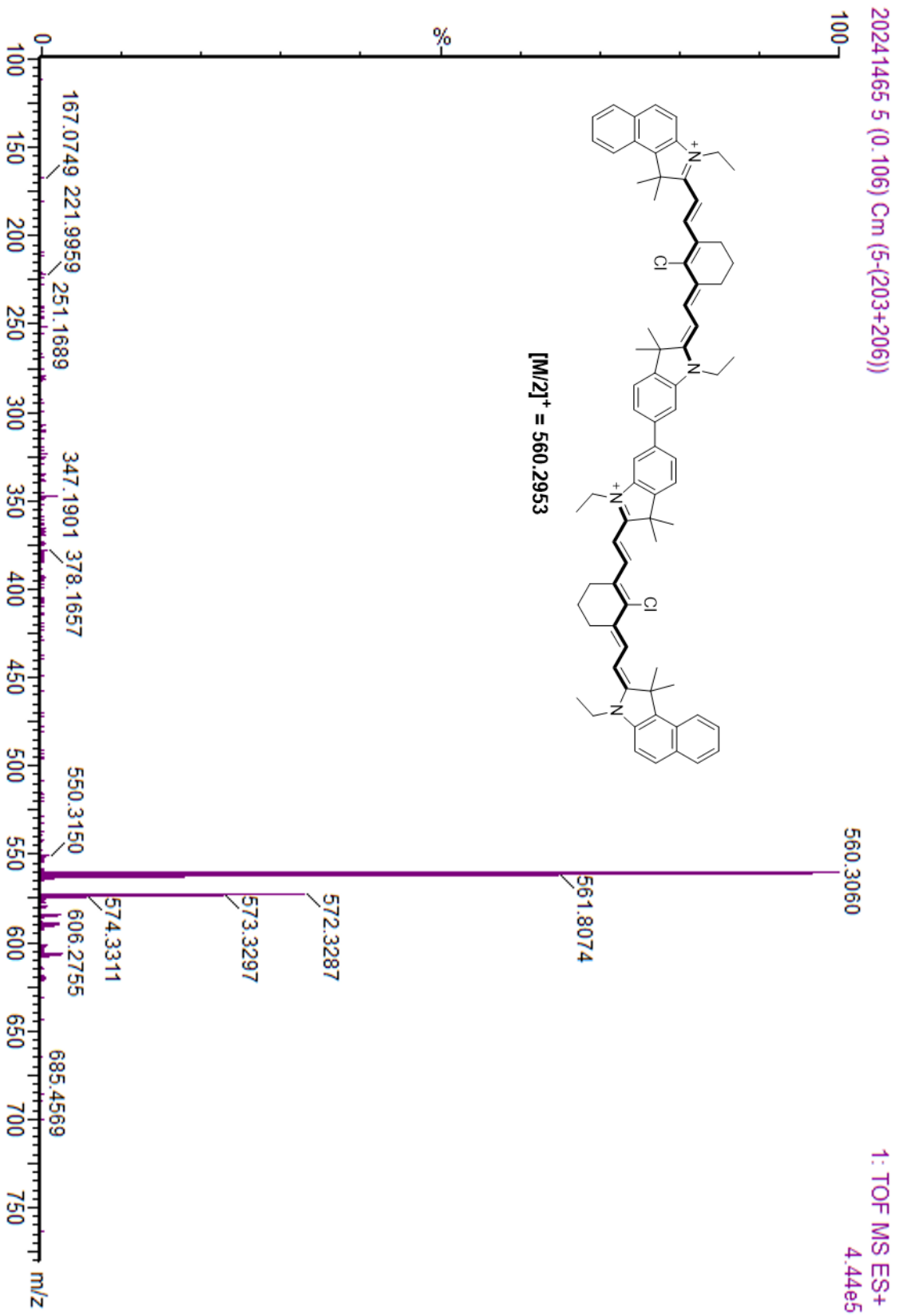

Figure S29: HRMS-ESI spectrum of **CyD7.5**.

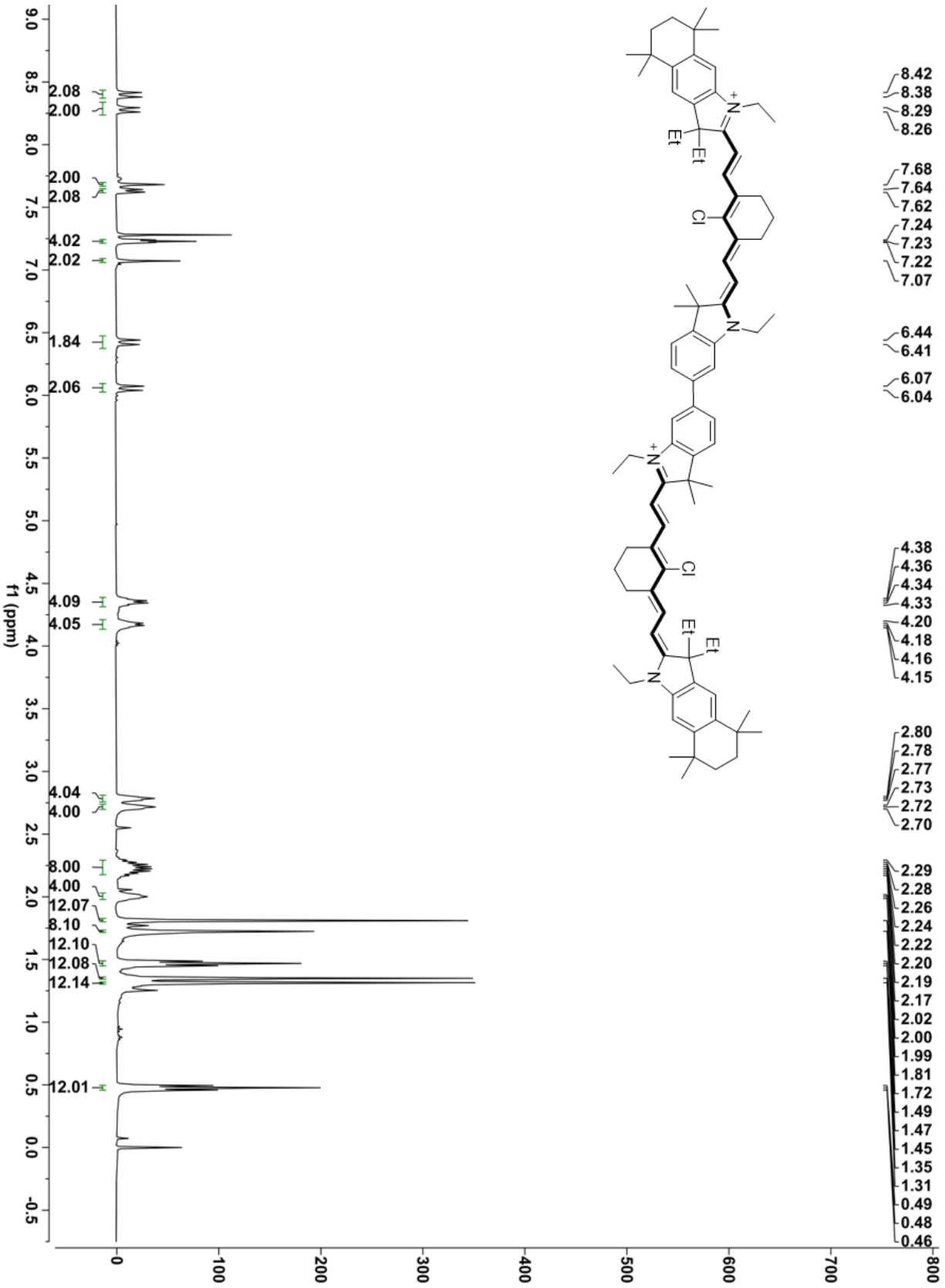

Figure S30: ^{1}H NMR spectrum of **CyD7Et** in $CDCl_3$ (400 MHz).

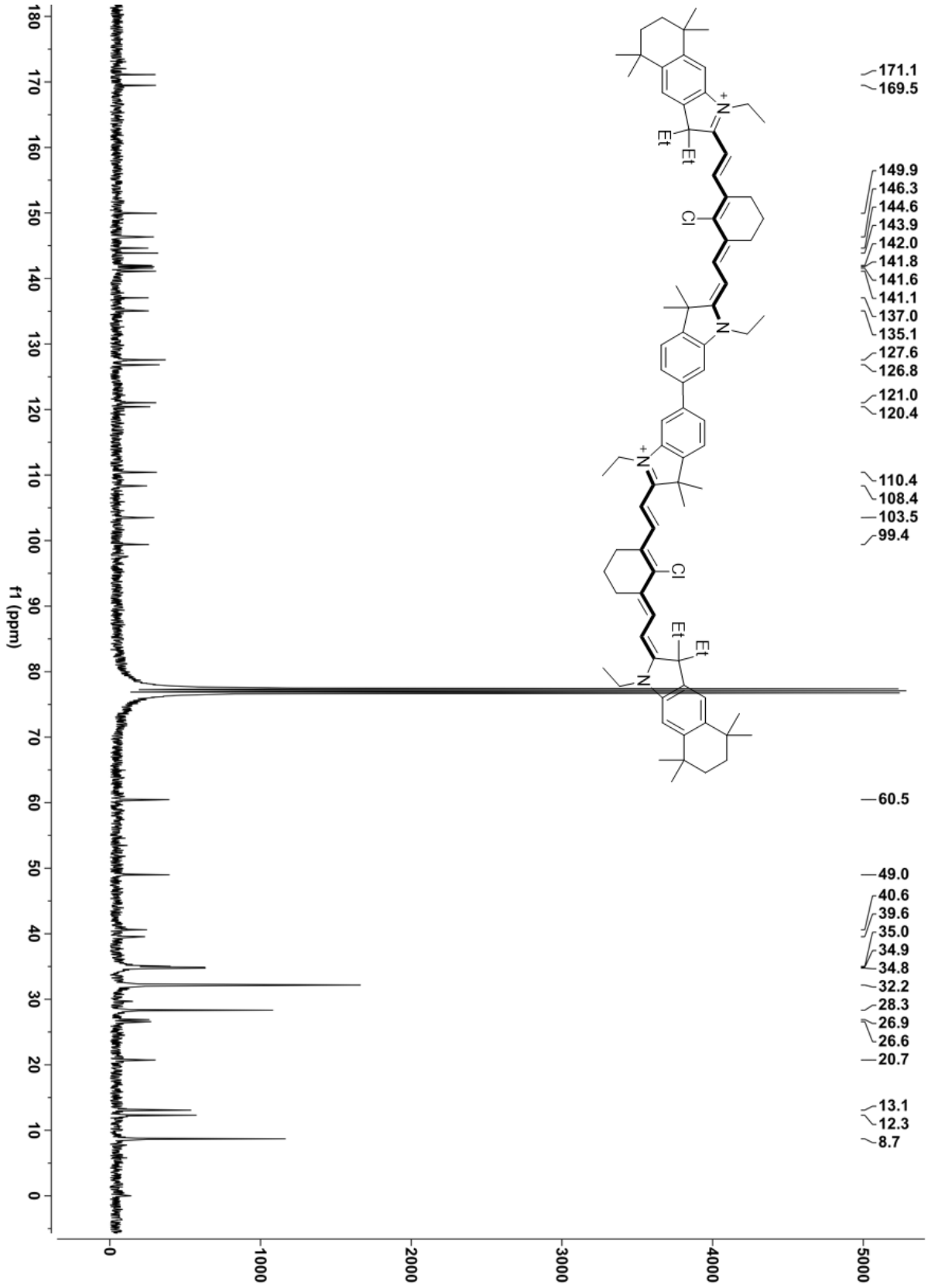

Figure S31: ^{13}C NMR spectrum of **CyD7Et** in $CDCl_3$ (100 MHz).

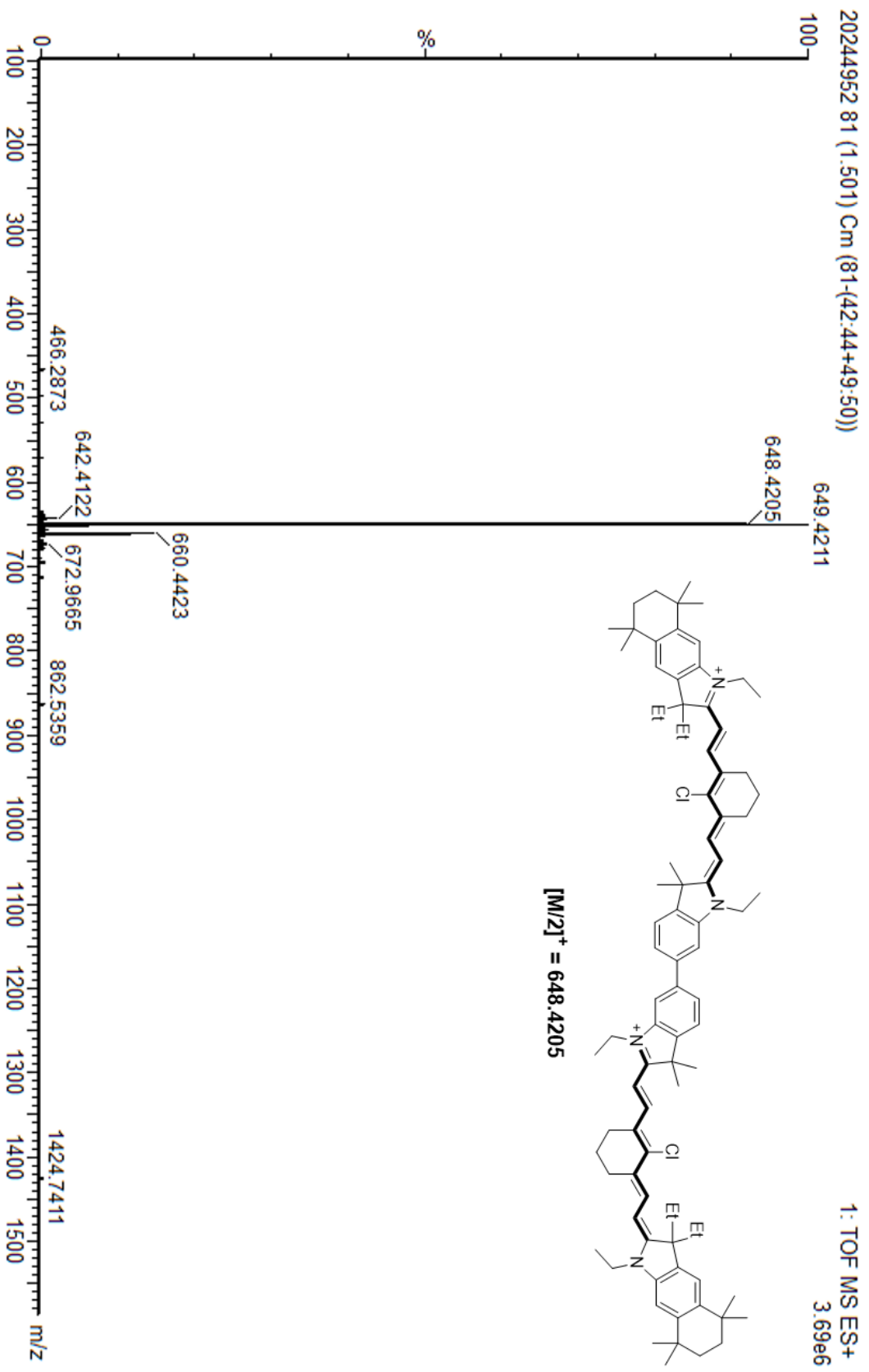

Figure S32: HRMS-ESI spectrum of **CyD7Et**.

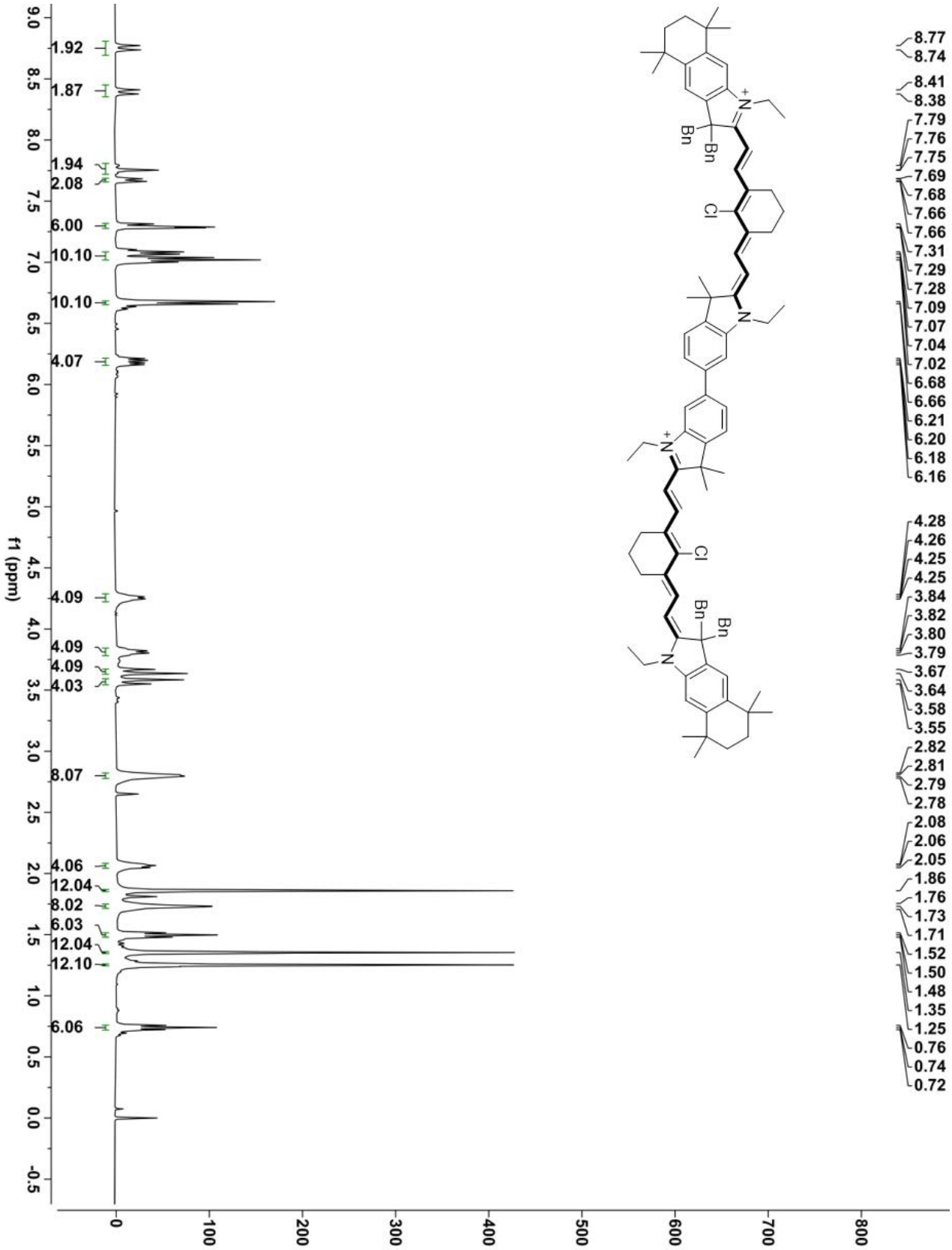

Figure S33: ^{1}H NMR spectrum of **CyD7Bn** in $CDCl_3$ (400 MHz).

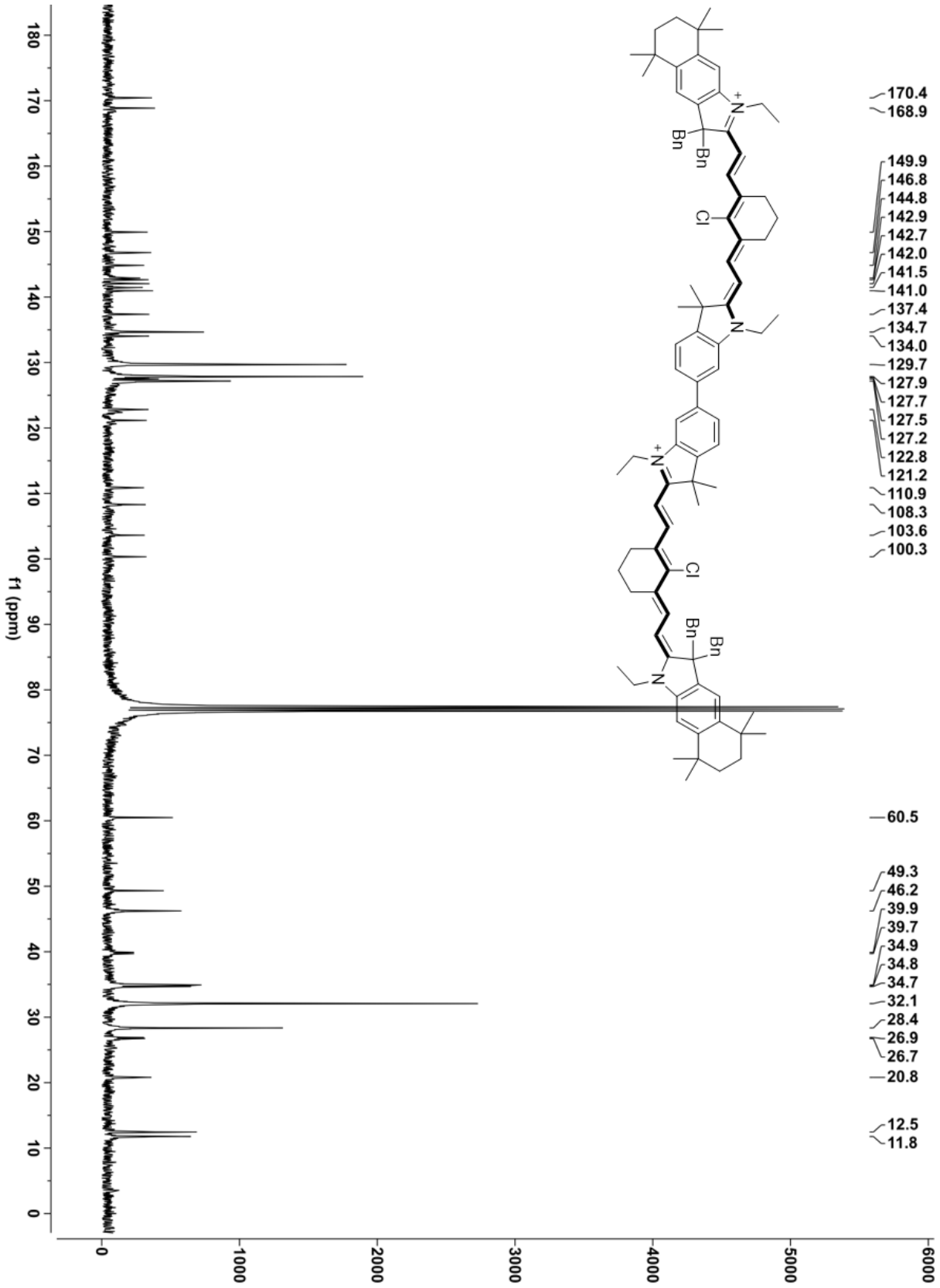

Figure S34: ^{13}C NMR spectrum of **CyD7Bn** in $CDCl_3$ (100 MHz).

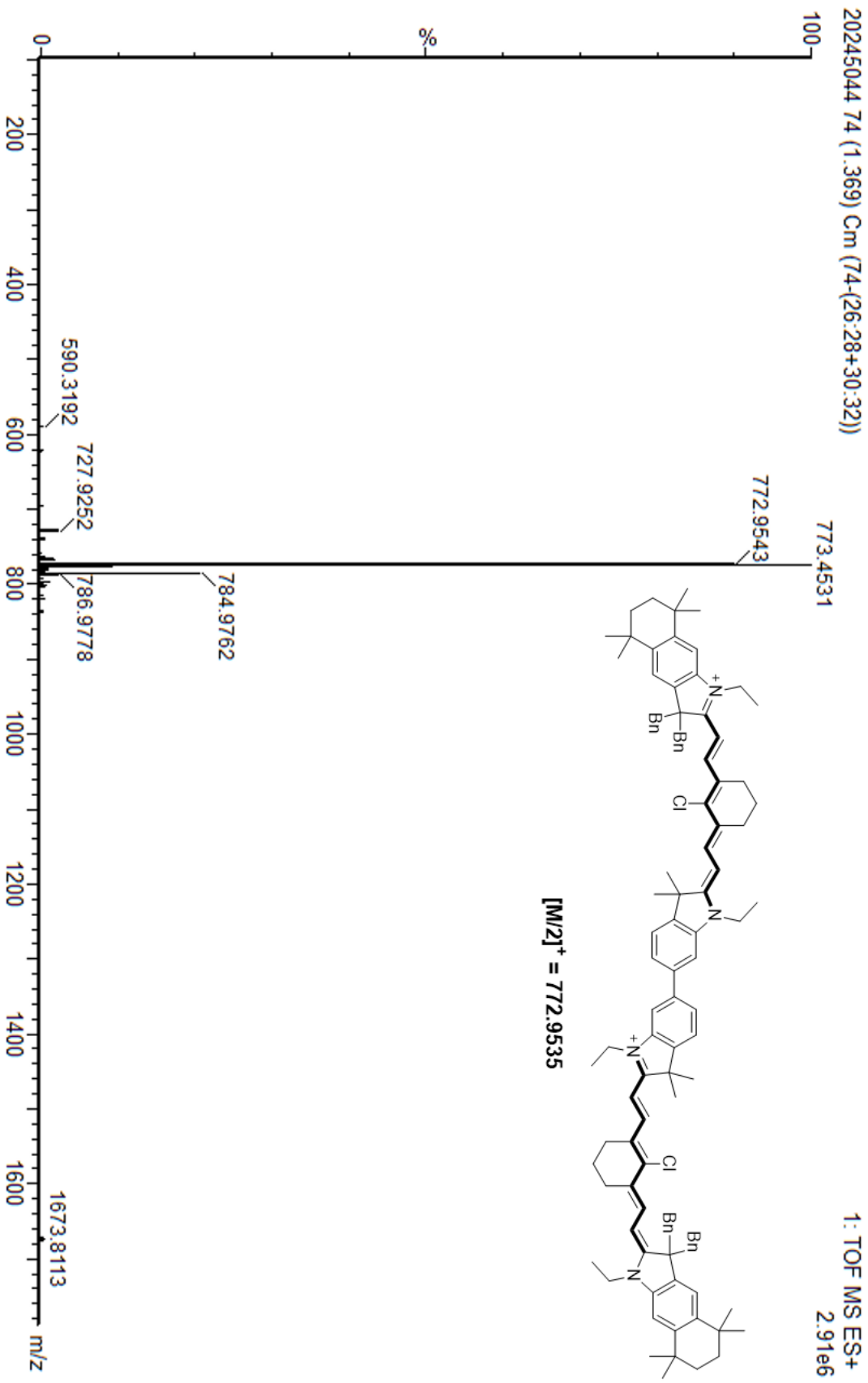

Figure S35: HRMS-ESI spectrum of **CyD7Bn**.

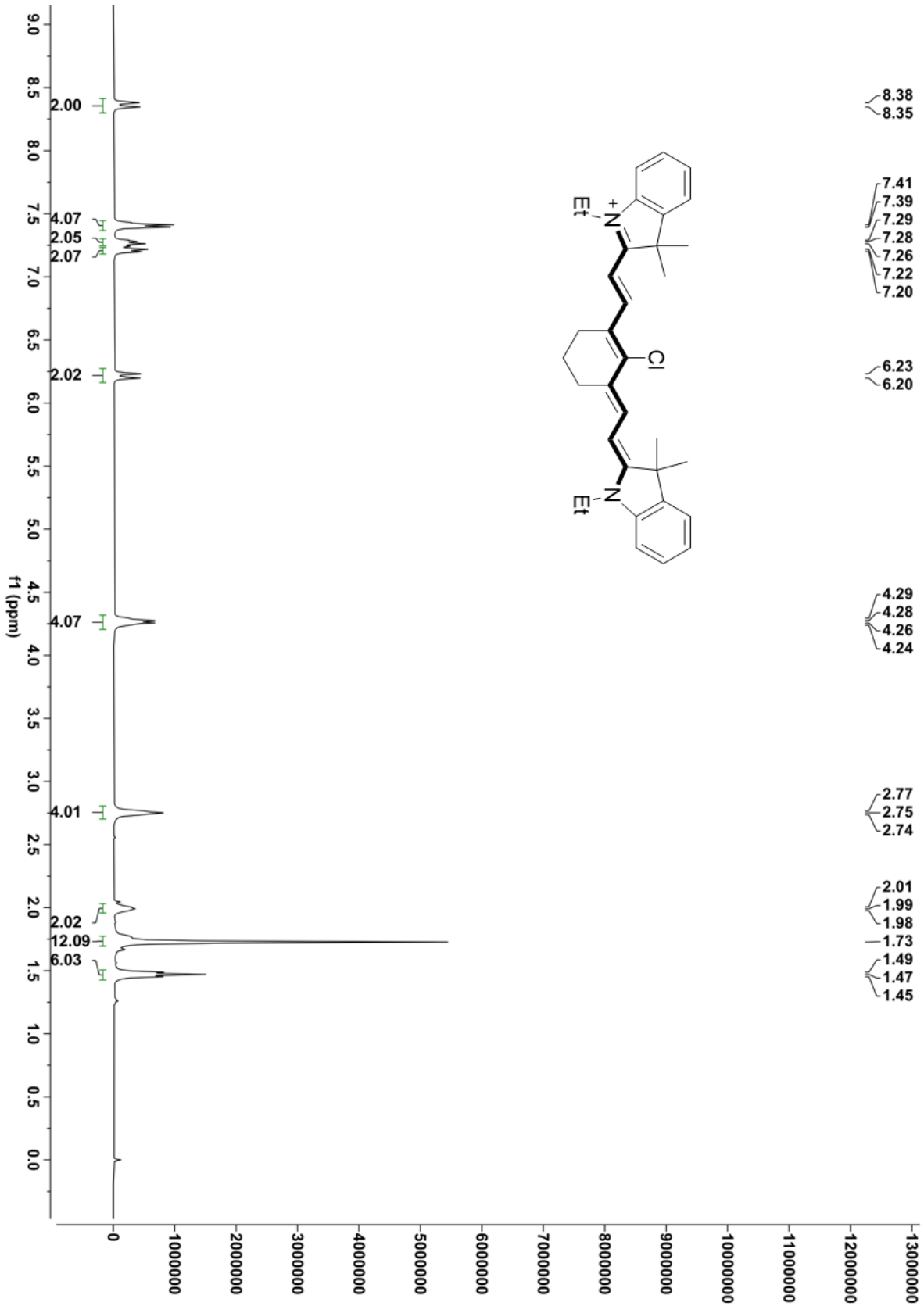

Figure S36: ^{1}H NMR spectrum of **M780** in $CDCl_3$ (400 MHz).

References

1. Liu, W.; He, S.; Ma, X.; Lv, C.; Gu, H.; Cao, J.; Du, J.; Sun, W.; Fan, J.; Peng, X. *Angew. Chem. Int. Ed.* **2024,** *63*, e202411802.

2. Zhang, Y.; Wang, B.; Yan, Q.; Lu, X.; Zhang, X.; Yao, C.; Qi, Y.; Yang, M.; Ge, G.; Qian, X.; Luo, X.; Yang, Y. *J. Am. Chem. Soc.* **2025,** *147*, 40739–40750.